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MAY 31, 1948
VOLUME 37, No. 11

AMERICAN DYESTUFF REPORTER

OFFICIAL PUBLICATION
of the
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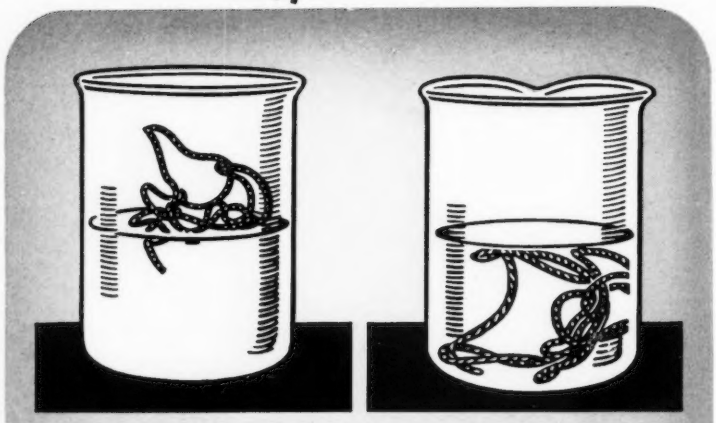

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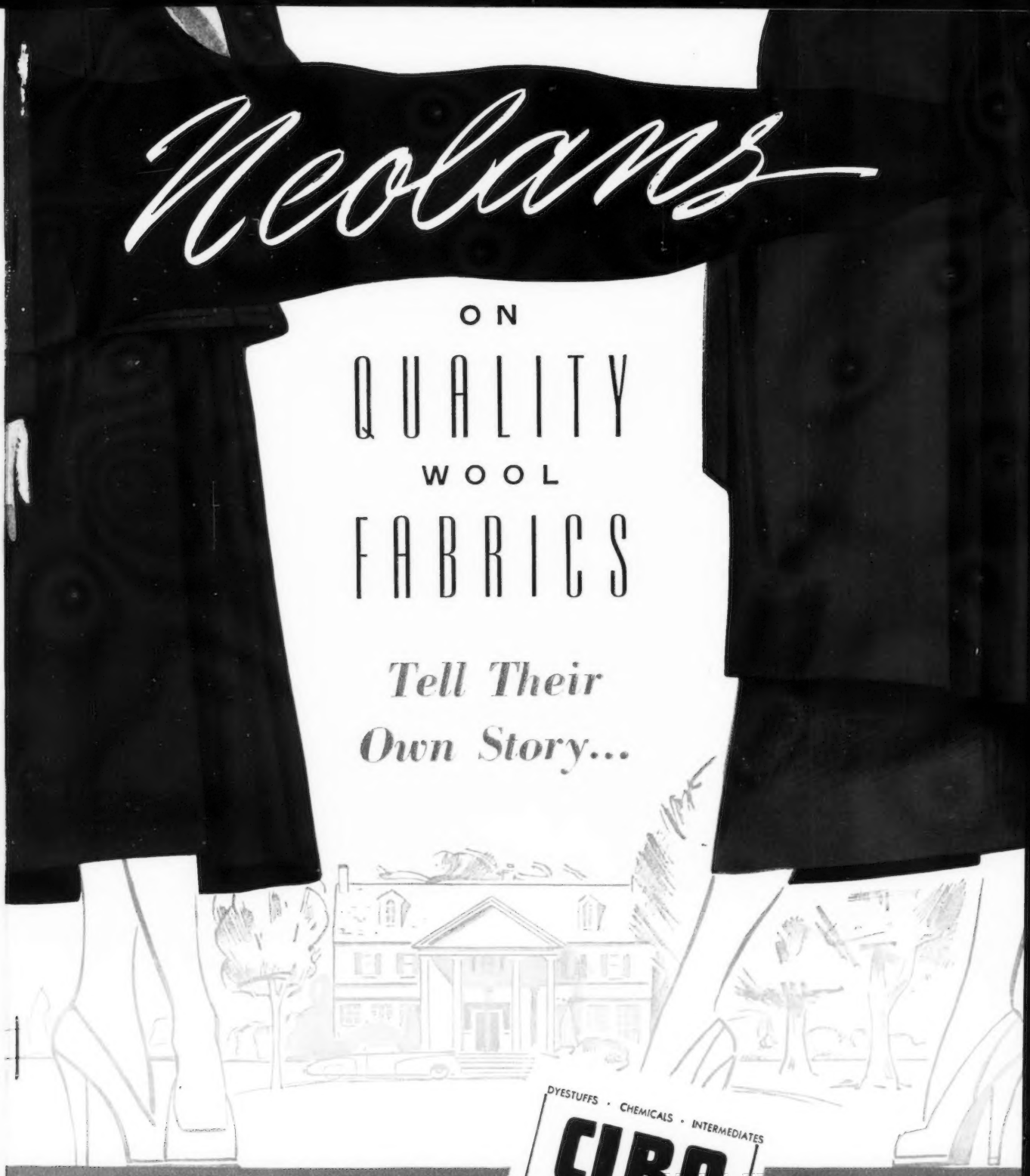

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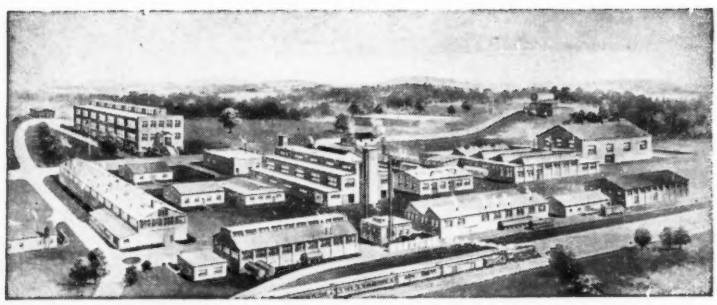

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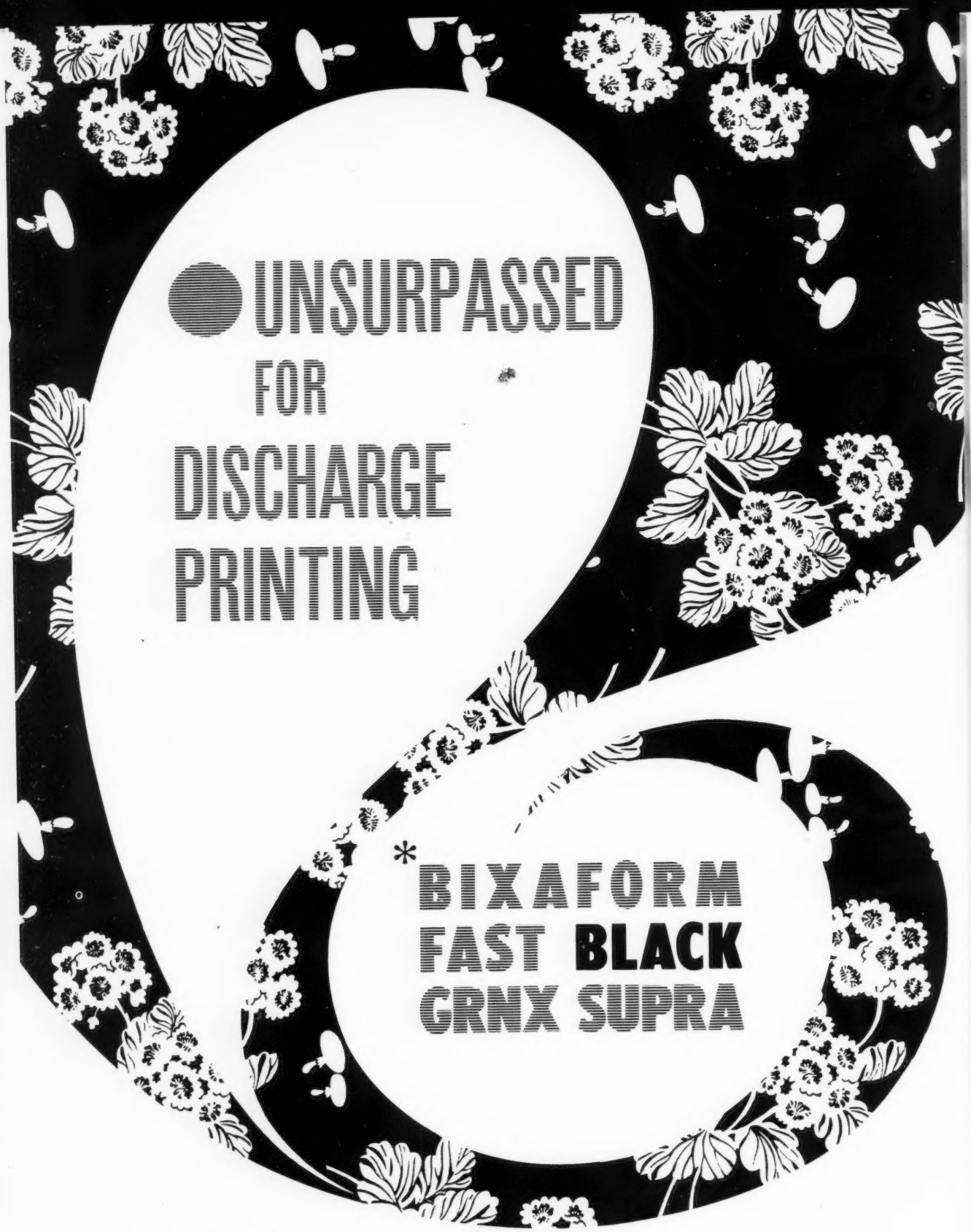

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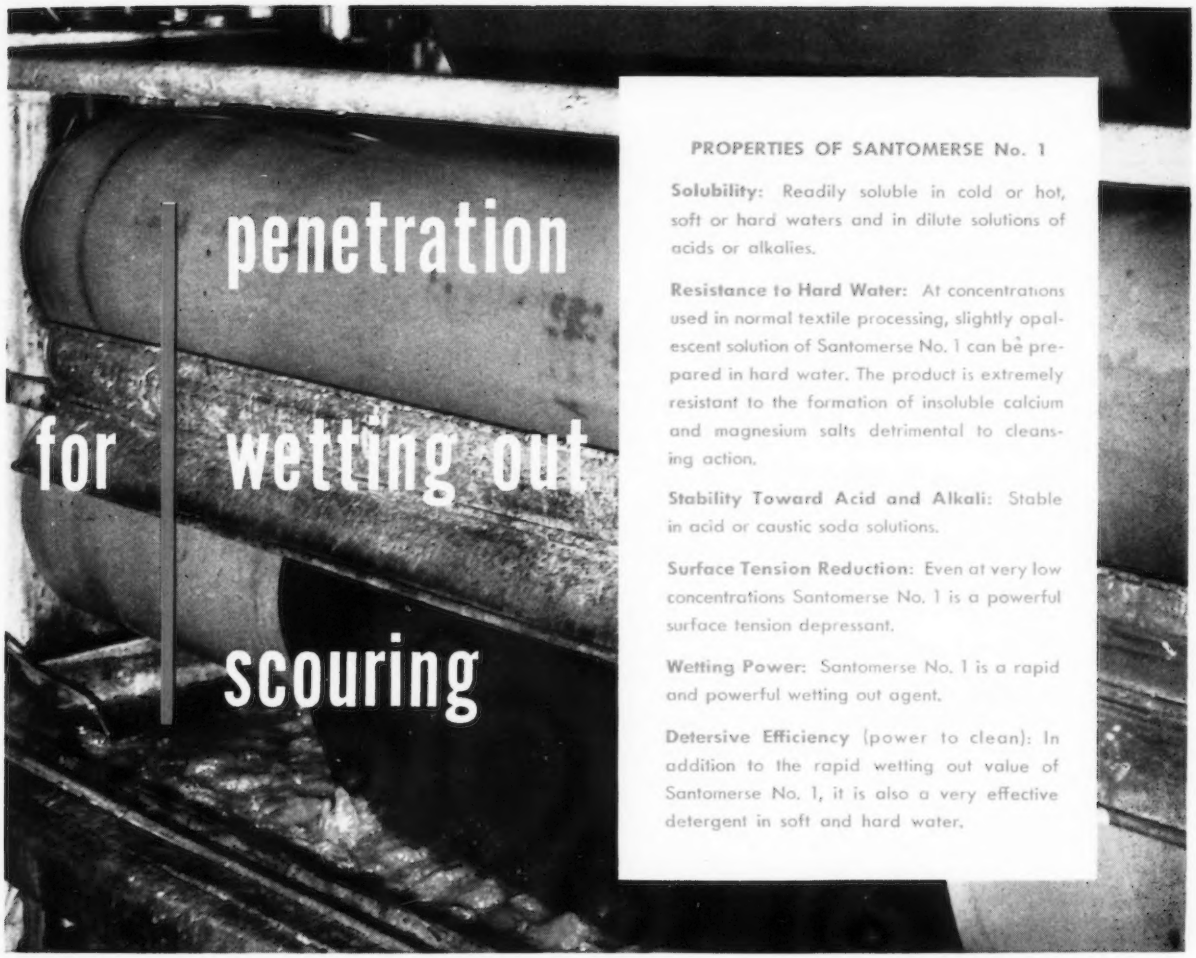

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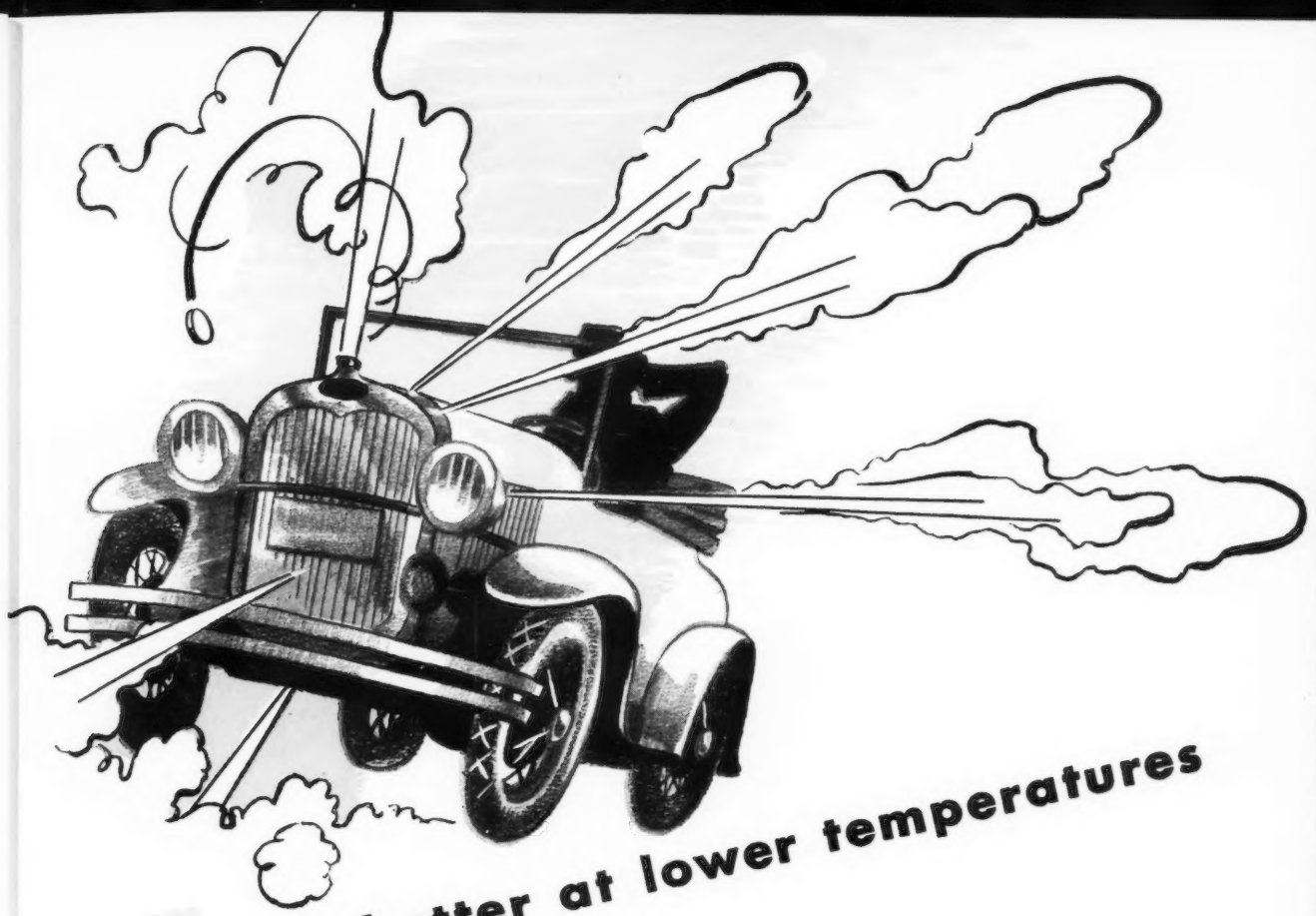

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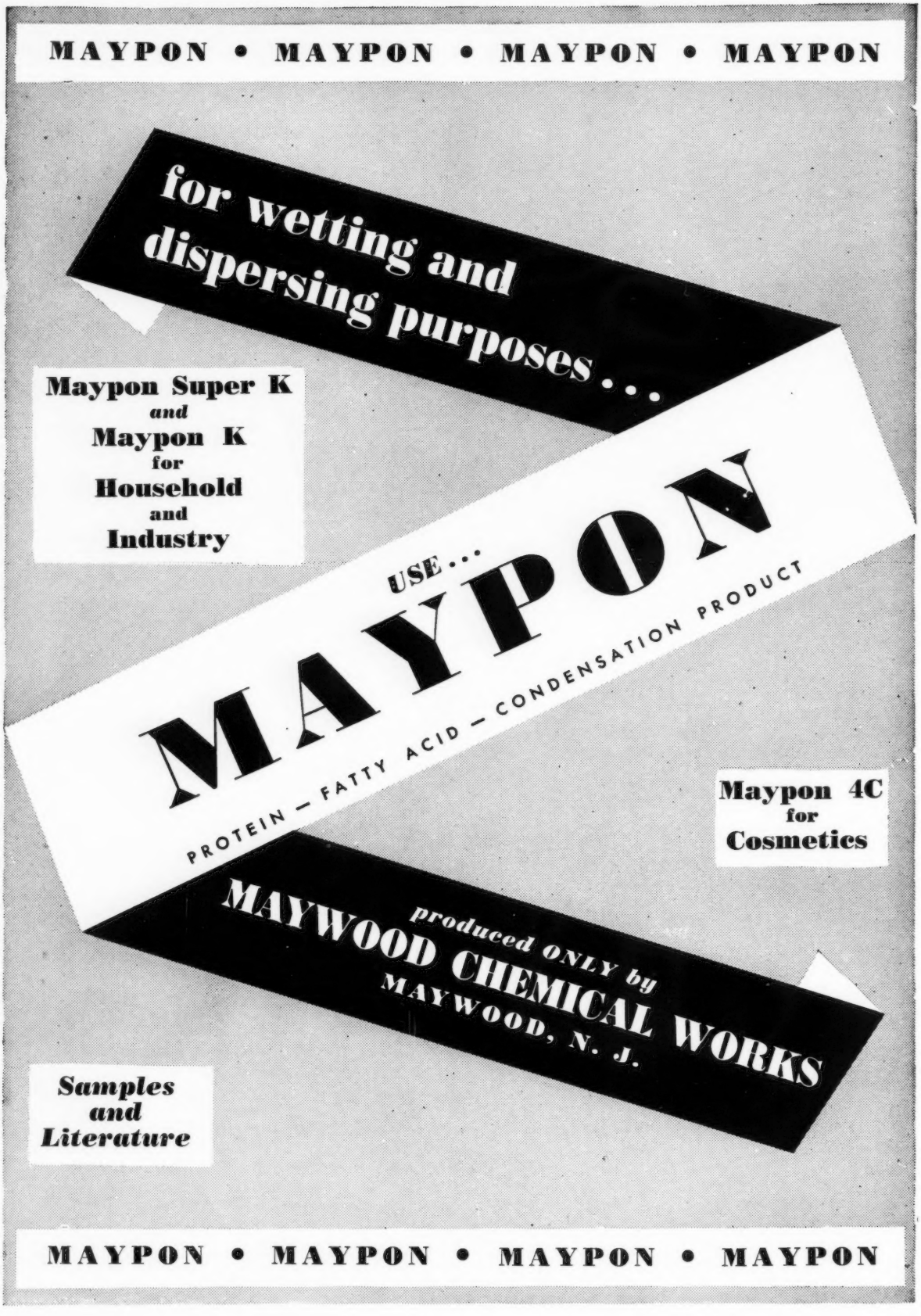
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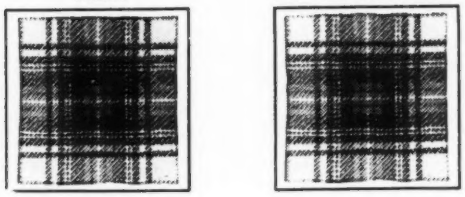
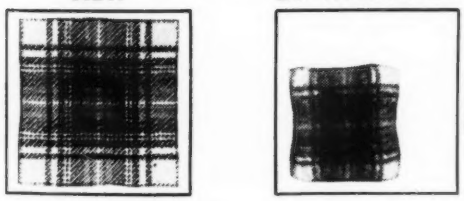


Untreated **WOOL**

NEW **20 WASHINGS**



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May 31, 1948

VOLUME 37 NUMBER 11

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May 31,

AMERICAN
DYESTUFF REPORTER

VOLUME 37 MAY 31, 1948 NUMBER 11

Development of Controlled Equipment for Testing Fastness to Atmospheric Gases

FRANCES KARR RAY*
Ellen H. Richards Institute,
The Pennsylvania State College

Introduction

A REPORT by Seibert in 1940 *(4)* and further data from a study at the Ellen H. Richards Institute *(3)* have shown the importance of controlled gas fading equipment both for achieving consistent reproducibility in test results and for determining means of accelerating the gas fading test.

It was the purpose of the present investigation to develop such modifications and additions to a current type of gas fading oven as might be necessary for control of the following factors:

1. Rate of air flow
2. Type of destructive gas
3. Quantity of destructive gas
4. Temperature
5. Humidity

Developmental Procedure

Description of Gas Fading Chamber Previous to Application of Controls

The gas chamber to which the controls were attached was built according to a blueprint provided by the American Association of Textile Chemists and Colorists with a few modifications, as described by the author in a separate report *(3)*. It was made of galvanized iron; it was provided with a two-inch inlet pipe to permit the entrance of gases from a lighted Meker-type burner. Two baffle plates inside the oven served to deflect the gases, which were kept in circulation by means of a four-bladed propeller driven by an electric fan motor, and which were emitted from the oven through an exit pipe fitted with a reducer. Calibrated dry and wet bulb thermometers, and a small glass water container for keeping the wick moist were fitted into a stopper and placed in an opening made in the top of the oven. These were used for checking the temperature and humidity in the oven.

An air-tight door consisted of a brass frame fitted with two pieces of double-thickness window glass placed approximately one-fourth inch apart, and provided with a screw-type of latch. The dead air space between the two pieces of glass provided insulation. The door was insulated further with two thicknesses of asbestos paper fastened on the outside. The top, sides, and back of the oven were covered with a layer of asbestos approximately one inch thick.

A Fischer and Porter calibrated rotometer was installed in the gas line so that the flow of gas could be controlled manually at the desired number of cubic feet per hour.

Control of Rate of Air Flow

The use of the auxiliary equipment planned for temperature and humidity control necessitated a means for drawing the air and gas mixture through the apparatus, and a method of controlling the rate of flow. Suction created by a water pump and manual control were used. A manometer-type of flowmeter was made and calibrated by means of a wet meter, for the purpose of measuring the rate of air flow. The working range of the flowmeter as calibrated, using water as the liquid, was 5.0 to 14.5 cubic feet per hour.

In order to dehydrate the air before it passed through the flowmeter, thus preventing condensation in the capillary, and also in order to bring the temperature down about to room temperature, a cold water condenser was installed between the oven exit pipe and the flowmeter.

In order to obtain a rate of flow comparable to that in the uncontrolled apparatus described in a previous report *(3)*, it was necessary to approximate the rate of flow prevalent when movement of air was effectuated by natural convection only. This was accomplished by the following means:

1. By setting the gas consumption at 3.2 cubic feet per hour (the rate which had been used in previous experiments);
2. by varying the rates of air flow; and
3. by noting the oven temperature after one-half hour at each rate.

At 11 cubic feet per hour the oven came closest to the usual temperature maintained in the uncontrolled apparatus. This rate was, therefore, assumed to be approximately the rate prevalent in the uncontrolled equipment, and was adopted for this study.

Quantitative Analysis of Oven Gases

In preparation for investigations involving close control both of the type and quantity of destructive gases, analyses of the quantities of oxides of nitrogen and of sulfur in the uncontrolled test apparatus (with use of a gas burner and wire gauge) were undertaken.

Oxides of Nitrogen. The phenoldisulfonic acid method was used for determining the proportion of oxides of nitrogen present in the oven gases in parts per million. The procedure was a modified form of that reported by Beatty, Berger, and Schrenk *(1)*.

In application of this method to the

* Deceased, March 1, 1947.

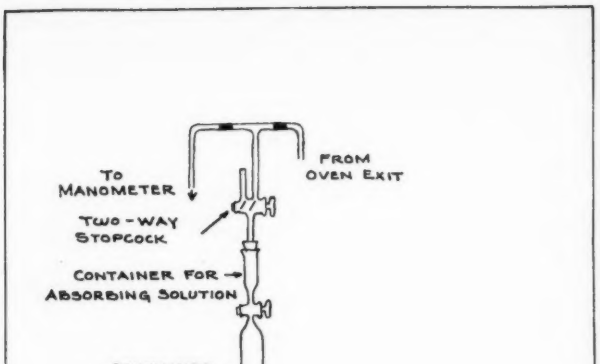

Figure 1
Sampling Tubes and Accessories.

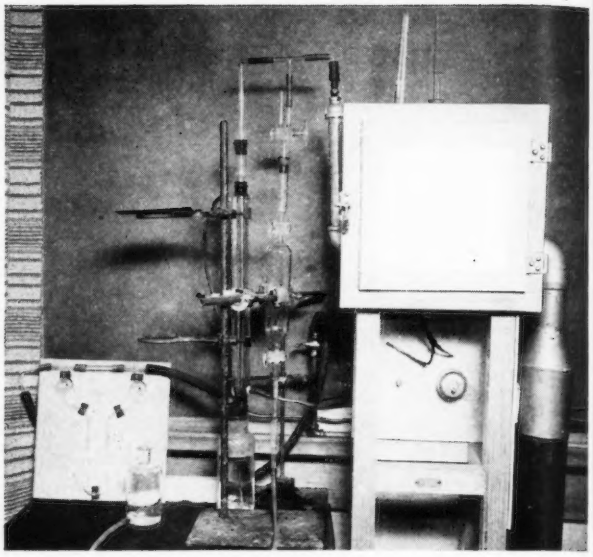

Figure 2
Gas Fading Oven with Complete Gas-Sampling Assembly.

analysis of gases in the gas-fading oven, a special technique was adopted for collecting the sample. Sampling tubes had a capacity approximately of 200 c.c. The exact capacity of each was determined by measuring the volume of water held when full. Each tube was provided with a stopcock at each end, and with a container at the upper end for the absorbing solution.

At the time of sampling, the gases were drawn through the oven by means of suction at the rate of 11 cubic feet per hour. A glass tee and a two-way stopcock were insterted between the oven exit and the flowmeter. The upper end of the sampling tube was attached thereto. To the lower end of the sampling tube a 10-c.c. leveling bulb, a three-foot length of rubber tubing, and a 250-c.c. leveling bottle were attached, as shown in Figure 1.

All connections between the oven exit and the leveling bulb were glass-to-glass.

Supplementary apparatus consisted of a ring-stand, two clamps and wire gauze to provide different levels for setting the leveling bottle during the course of the procedure.

A photograph of the oven with complete gas-sampling assembly, condenser, and flowmeter is shown in Figure 2.

In preparation for drawing the sample, the leveling bottle was filled with ammonia-free distilled water, and was used as a means of creating a secondary vacuum.

At the time of sampling, the oven temperature, oven humidity, and atmospheric pressure were recorded.

The collection of the sample and the addition of absorbing liquid were accomplished in four major steps, which are summarized as follows:

1. The sampling tube was purged of air by bringing the water level to the two-way stopcock, which was then switched from air to gas line, allowing gas to fill the upper part of the sampling tube, as the leveling bottle was lowered.
2. After repeating the above procedure, this time bringing the level of gas slightly beyond the bottom of the sampling tube, the tubing was held with one hand and the other was used to shut the two-way stopcock.
3. The leveling bottle was raised so that its water level was slightly above the bottom stopcock of the sampling tube. A slight release of fingers holding the tubing allowed the water level to rise to the bottom stopcock, which then was shut. The two-way stopcock was opened to air momentarily. This gave a measured volume of gas at atmospheric pressure.

The above procedure was performed rapidly so that the temperature of the gas did not drop far below the oven temperature.

4. The absorbing liquid was poured into the top container. The bottom stopcock was opened and approximately 10 c.c. of gas was drawn into the bulb by lowering the leveling bottle. The bottom stopcock was closed and the leveling bottle immediately was set at a high level to place the withdrawn gas under pressure. The top stopcock was opened, letting the solution enter the tube, and immediately was closed. The bottom stopcock then was opened, and the water level was allowed to rise *thereto without stopping*. At this point, the water was stopped at once by pinching the tubing; and then the stopcock was closed.

The procedure for analysis was in accordance with that specified by Beatty, Berger, and Schrenk *(1)*, with the exception of the method of transferring the absorbing liquid from the sampling tube. This was accomplished first by opening both stopcocks, pouring the solution into a 150 c.c. beaker, and then attaching a short piece of rubber tubing to the lower end of the tube, which was placed in a horizontal position. The tube was rinsed with three 30 c.c. portions of ammonia-free distilled water, closing the stopcocks after each addition, so that the water could be rinsed thoroughly over the sides of the tube. The washings were added to the absorbent in the beaker.

A blank and three standards were pre-

pared at the time of sampling. Nessler tubes were used for matching the colors of the samples with reference to that of the blank and standards. In using this method of color comparison, the author was unable to distinguish differences of less than five parts per million. Since this amounted to an error of about 25 per cent corrections were not applied for exact volume of sample, temperature, pressure and humidity, all of which would have amounted to much less than the above error.

Because visual matching of color intensity is very difficult with the pale yellows encountered in this procedure, it is believed that a desirable degree of accuracy can not be obtained without the use of a suitable photoelectric colorimeter.

Three samples of gas taken at hourly intervals over a period during which the gas consumption fluctuated between 3.0 and 3.4 cubic feet per hour were found to contain approximately 20 parts per million by volume of oxides of nitrogen.

Oxides of Sulfur. An attempt was made to analyze the oven gases for oxides of sulfur (sulfur dioxide and sulfur trioxide), following a modified version of the titration method described by Thomas and Abersold *(5)*. This attempt was unsuccessful because of the minute quantities of the above gases present, and because of the interference of the carbon dioxide in the gas.

Description of Equipment for Control of Temperature and Humidity

In order to study the effect of different relative humidities on gas fading, it was necessary to provide a means of introducing moisture into the gas-fading chamber, as well as a means of removing it, so that controlled humidity conditions would be investigated. A study of the effect of different temperatures as a means of reheating the air after moisture adjustment also was required.

A diagram of the gas-fading chamber with the equipment designed and built for temperature and humidity control is shown in Figure 3.

As shown in the diagram, the arrangement was such that the air and gas mixture coming from the burner could be led through the apparatus designed to adjust the humidity before the final temperature adjustment.

In order to offset potential small leaks which would permit air to be drawn from sources other than the gas and air inlet, a blower was installed in the inlet pipe at a point above the burner shield. Its function was to build up pressure in the air pipes and in the oven, so that the air drawn through would come from this

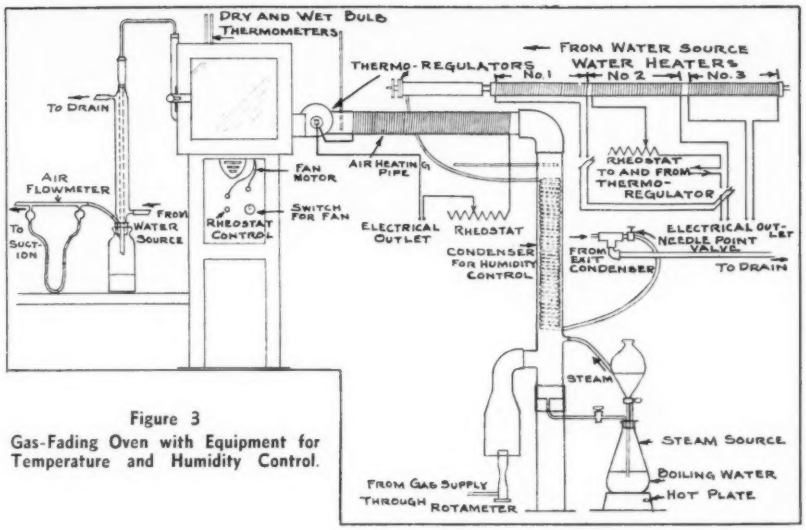

Figure 3
Gas-Fading Oven with Equipment for Temperature and Humidity Control.

source rather than through leaks. The blower consisted of an eight-bladed propellor which fitted closely to the inside circumference of the pipe, and which was turned by an electric fan motor modified for use in an inverted position.

The equipment for moisture adjustment consisted of a steam source, a water condenser or cooler, and an electrical heating system for the water. Before it passed through the condenser, the water was to be heated electrically to the temperature at which the counterflowing saturated air would have the desired amount of moisture.

As a source for steam, water was heated over a 550-watt hot plate in an Erlenmeyer flask provided with a rubber stopper into which were fitted a separatory funnel (through which water could be added) and two tubes leading to and from points below the condenser. The lower end of the pipe containing the condenser was closed off and the tube leading from it back to the steam source was provided with a stopcock so that the condensate could be returned to the flask at a constant rate. Although a hot plate with adjustable temperatures would have been more desirable than the single-heat plate which was used, the amount of steam could be adjusted to a certain degree by raising or lowering the flask.

The water condenser consisted of three-sixteenths-inch copper tubing leading from the water source to the drain, and wound spirally with coils three-sixteenths of an inch apart, to a length of 18 inches and a diameter of about 1½ inches. This was mounted in a 30-inch length of 2½-inch brass pipe so that the condenser coils were about one-half inch from the inside of the pipe. A needle-point value, placed between the condenser and the drain, permitted adjustment of the rate of flow of the water, which in turn affected the efficiency of the condenser, thereby providing an additional means of varying the humidity. A thermometer well of German silver placed above the condenser was provided in order that the approximate temperature of the saturated air could be read.

A 38-inch length of copper tubing with an overall diameter of 1.375 inches and a wall thickness of 0.065 inches led from the water source to a nine-inch-long brass cylinder in which a thermo-regulator was mounted and to which the condenser was attached. The copper tube was wrapped with a layer of asbestos paper and then wound with three separate lengths of Nichrome resistance ribbon wired in parallel and providing three different water-heating units.

Heaters labelled Numbers 1 and 3 in the diagram were made of 190-inch lengths of 0.490-ohm-per-foot resistance ribbon three sixteenths of an inch wide, wound with the coils approximately one sixteenth of an inch apart. These two heaters carried a current of 14 amperes and had a heating capacity of 1540 watts each.

Heater Number 2 consisted of a 190-inch length of 0.618 ohm-per-foot resistance ribbon of the same width and spacing as the other heaters. It carried a current of 11 amperes and had a 1210-watt maximum heating capacity. This heater was made adjustable by connecting it in series with a rheostat (with 2.8 ohms of resistance and a 12.3-ampere capacity) and a bimetallic immersion type thermo-regulator (with a temperature range of —50 to 400° F. and a contact rating of 10 amperes).

Heaters 1 and 2 were connected to the

same electrical outlet, but each was provided with a double-pole, single-throw switch. Heater Number 3 was connected to a separate outlet. Therefore, any one or all heaters could be used as desired.

After the moisture content was adjusted, the air had to be reheated to the desired temperature. For this purpose, an air-heating unit was made by insulating a 22-inch length of two-inch brass pipe with one layer of asbestos paper, and then winding Nichrome resistance ribbon over an 18-inch length of the pipe. The ribbon was three sixteenths of an inch wide and the coils were spaced slightly less than one sixteenth of an inch apart, so that a length of 48 feet was used.The resistance of the ribbon being 0.228 ohms per foot, a total resistance of 11 ohms provided for a current of 10 amperes and a heating capacity of 1100 watts. For the sake of flexibility, this heater was connected in series with a rheostat carrying 47 ohms of resistance with a 3.1-ampere capacity. A bimetallic type thermo-regulator with a temperature range of —50 to 400° F. and a contact rating of 10 amperes was installed lengthwise in a short length of two-inch brass pipe between the heater and the oven. The thermo-regulator was connected in series with the resistance of the air-heater. A thermometer well installed in the air pipe at a point 1½ inches beyond the air heater permitted measurement of the temperature at this point.

The water heaters, the condenser pipe, and the air heater and thermo-regulator pipe were covered with magnesium insulation. In addition, a one-half-inch thickness of asbestos insulation was added to the air heater and thermo-regulator pipe, making a total of 1½ to 2 inches of insulation on these areas.

In order to maintain the desired temperatures, it was necessary to provide heaters for the oven itself. Two 8 x 1½-inch 250-watt strip heaters were attached on the outside of the oven across the middle portion of the exit side and the back (underneath the insulation). They were wired in such a manner that they could be used separately for 250-watt heating capacity, in series for 125 watts, and in parallel for 500 watts. In addition, a 3.0 ampere, 47-ohm rheostat was connected in series with the side strip heater. The apparatus then could be heated rapidly, and the temperature maintained at any practical level. A bimetallic-type of thermo-regulator, with a temperature range of 60 to 250° F. and a contact rating of 35 amperes, was wired in series with the strip heaters and the sensitive bulb inserted in the oven through a Number 5 rubber stopper placed in an opening in the center of the top.

A schematic diagram, switch board diagram, and switch combination for various heats are given in Figure 4.

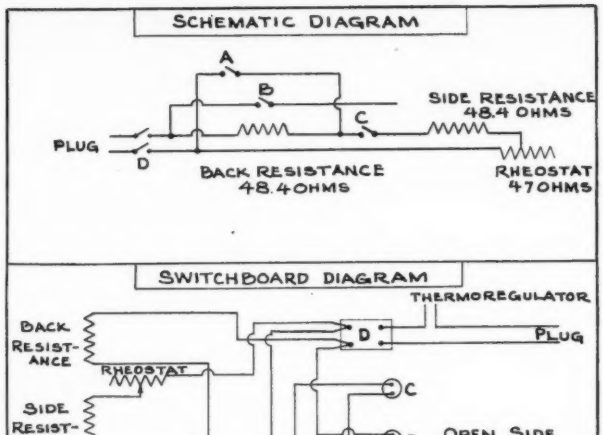

Figure 4
Oven Heating Unit.

Equipment Tests

Pressure in Oven

A water-filled manometer was fitted into a stopper placed in the center hole in the top of the oven, and the blower was turned on together with suction sufficient to give an air flow of 11 cubic feet per hour. The pressure in the oven was found to be very slightly greater than atmospheric. Theoretically, this was just sufficient to offset leaks without forcing air and gas out through them.

Tests on Oven Heaters

In spite of the fact that adjustment of the wattage of the oven strip heaters was regulated to the approximate heating capacity needed, it was found that the temperature range of the thermo-regulator varied to the extent of ±4.5° C. For this reason, supplemental manual control was necessary when a limited temperature range and dependent relative humidity range were desired. It should be noted that, particularly at high humidities, a fluctuation in temperature will result in a much greater fluctuation in relative humidity for a given moisture content in the air.

Tests on Humidity Apparatus

Upon testing the humidity apparatus with oven temperatures of 55 and 65° C., it was found that a wide range of relative humidities—from 15 per cent to 70 per cent or more—could be obtained either with or without the use of the burner. The apparatus, however, presented several handicaps. With the oven temperature at 65° C., high relative humidities caused moisture to condense on the inside of the oven door and on the capillary of the flowmeter, in spite of the cold water condenser which preceded it. The condensation on the door was eliminated when the relative humidity was brought down to 55 per cent or less, but the condensation in the capillary of the flowmeter still prevented accurate control of the air flow at this humidity range.

A comparison of the approximate condenser water temperature (measured at the drain) with the approximate temperatures of the saturated air passing over the condenser revealed that this part of the

apparatus was not 100 per cent efficient, even with the water passing through the condenser at its maximum rate of flow (one liter in 40 seconds). The saturated air temperatures in the condenser pipe both for high and low relative humidities in the oven were found to be about 8 to 10° C. higher than the corresponding water temperatures. This, however, was not found to be a severe handicap.

It was found also that the amount of heat to be applied to the water for a certain humidity at a given temperature varied from time to time, possibly depending on the pressure and the temperature of the water before heating, and on the room temperature and humidity.

Where heater Number 2 was used, either alone or in combination with one or both of the other water heaters, the water temperature control could be made automatic by the use of the thermo-regulator. The amperage rating of the thermo-regulator and rheostat did not permit their use with heaters 1 or 3. In practice there were times when heater Number 2 could not be used to achieve the desired humidity, and the other heaters had to be controlled manually. This indicates the desirability either of a thermo-regulator or of a rheostat with an amperage rating sufficiently high to permit it to be connected in series with each of the three heaters, or with heater Number 1 or 3.

Use of Burner With Air-Conditioning Apparatus

After suspending six specimens of cellulose acetate satin dyed with 1.0 per cent of Celanthrene Brilliant Blue FFS in the oven for a total period of 16 hours, during which time the gas consumption was kept at 3.2 cubic feet per hour, the temperature between 51.5 and 58.5° C., the relative humidity between 25 and 70 per cent, and the absolute humidity between 0.028 and 0.070 pounds of water vapor per pound of dry air, there was no apparent fading of the exposed specimens. During this test period no steam was entered into the apparatus, the moisture content of the gas being sufficient to provide a wide range of humidities. This indicated that the destructive gases were not reaching the oven.

Examination of the copper condenser showed that corrosion was taking place. Moreover, the condensate which returned to the steam source carried with it a copper-colored powder. Although both the ferrous sulfate and the diphenylamine tests for nitrate gave negative results, it was possible that the tested condensate collected over a four-hour period contained minute traces of nitrate.

An explanation of what probably occurred was that the major portion of the burnt gas fumes was oxidized to nitrogen dioxide or tetroxide upon contact with air, some of this gas then reacting with moisture to form nitric acid:

$$H_2O + 3NO_2 \rightarrow 2HNO_3 + NO$$
$$2NO + O_2 \rightarrow 2NO_2$$

The probable reactions of the nitrogen dioxide and the nitric acid with the copper in the condenser and pipe were as follows:

$$6\ Cu + 4NO_2 \rightarrow 6\ CuO + 2NO + N_2$$
$$3\ Cu + 8HNO_3 \rightarrow 3\ Cu(NO_3)_2 + 2NO + 4H_2O$$
(dilute)

An attempt to stabilize the equipment against further reactions of this type, by operating it with the burner for a total period of 25 hours was unsuccessful, that is to say, the test samples showed no fading.

The only alternative was to by-pass the copper condenser and as much of the brass pipe as possible by entering chemically generated gas at a point closer to the oven, then eliminating the burner. This plan involved the use of bottled nitric oxide, displacing it with water.

Adjustments

Use of Chemically Generated Oxides of Nitrogen With Air-Conditioning Apparatus

The thermo-regulator formerly intended for use with the air-heating pipe was eliminated, and the opening made there for (about five inches from the oven) was chosen as the most convenient point at which to enter the gas. An air-tight flange connection was made to fit the opening. Into this a 25-inch length of one-fourth inch stainless steel tubing was fitted by means of a compression union.

Preliminary experiments showed that the gas-fading test could be accelerated to any desired degree, depending upon the rate of flow of oxides of nitrogen into the oven. It was apparent that the use of the pure gas and very slow, uniform rates of gas flow were desirable for developing a controllable, reproducible test procedure.

The low solubility of nitric oxide in water made it desirable for the initial form of the gas to be used, although this gas was oxidized to nitrogen dioxide which in turn formed nitrogen peroxide in an equilibrium reaction.

Further experiments resulted in the development of the following pieces of auxiliary equipment, with methods for their use:

1. Equipment and method for generating pure nitric oxide;
2. Equipment and method for collecting bottles of sulfur dioxide;
3. Constant leveling devices for maintaining slow, uniform rates of flow of the above gases.

Equipment and Method for Generating Nitric Oxide

Nitric oxide was generated by the ac-

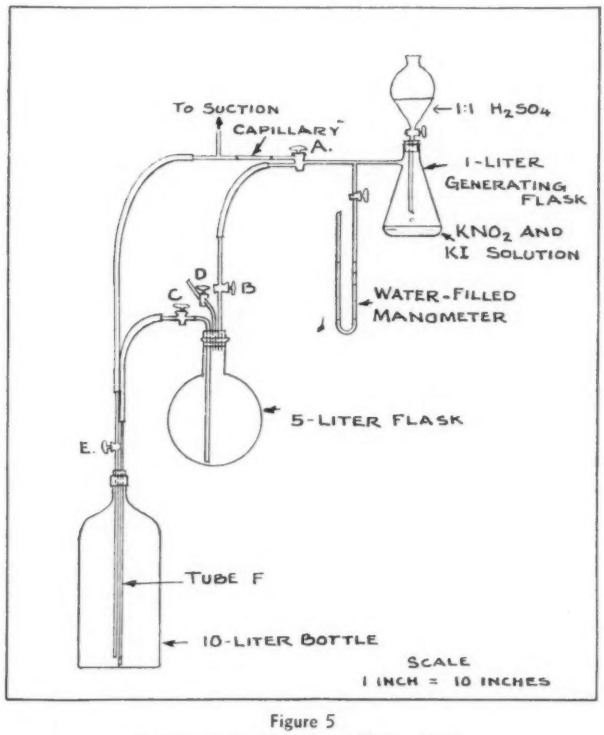

Figure 5
Equipment for Generating Nitric Oxide.

tion of 1:1 sulfuric acid (freshly boiled) on a mixture of one part of potassium iodide to two parts of potassium nitrite dissolved in freshly-boiled distilled water. This method was originally suggested by Winkler *(6)*, and later recommended by Greenspan *(2)* as producing pure nitric oxide. The series of reactions involved may be summarized as follows:

$$4H_2SO_4 + 2KNO_2 + 2KI \rightarrow 4KHSO_4 + 2NO + I_2 + 2H_2O$$

A diagram of the equipment used in this study for generating nitric oxide is given in Figure 5.

This equipment provided for displacing the water in an air-tight five liter flask with gas at approximately atmospheric pressure. All connections were wired and shellacked in order to render the apparatus as nearly air-tight as possible.

Preparatory to generating the gas, the bent tube containing stopcock D was filled with water, and the stopcock was closed, making certain there were no air bubbles. This tube was intended for use with the constant level device, only, and was not employed in the procedure for preparing the gas. The method of procedure for the latter consisted of the following steps:

a. The solution of potassium nitrite and potassium iodide was poured into the generating flask. With the stopcock closed, the acid solution was poured into the separatory funnel.

b. The five-liter flask was filled with distilled water. Then, with stopcock A closed, B, C, and E open, the suction was turned on. When Tube F filled with water, stopcocks E, C, and B were closed and the stopper on the 10-liter bottle was loosened. This prepared the siphon and helped remove the air between A and D.

c. The stopper was removed from the 5-liter flask, which was then refilled to the top with water. After the stopper was replaced, stopcocks B and C were opened.

d. With stopcock A partially open to the suction, the gas was generated by allowing the acid to drip slowly into the generating flask. The suction was left on for a few minutes (until the fumes were no longer brown) in order to displace the air in the flask. During this process the pressure was allowed to become slightly less than atmospheric.

e. Stopcock A was closed and the pressure was allowed to build up to slightly greater than atmospheric. A was then switched to the flask to be filled with gas.

f. As the gas entered the flask it displaced the water, forcing it into the 10-liter bottle. During this process the flow of acid was adjusted from time to time so that the pressure in the generating flask was kept slightly greater than atmospheric. (This precaution was taken in order to prevent air from entering the generating flask through possible leaks.)

g. When the five-liter flask was filled with the gas, stopcocks B and C were closed. The manometer was closed and two-way stopcock A was switched back to the suction until the gas was no longer being formed.

h. The 10-liter bottle was emptied by opening E to suction.

i. The edge of the stopper next to the flask and its tube connections were given a double coat of shellac to help prevent leaks. To offset the effect of small leaks, however, it was found advisable to make a new supply of gas at least once a month.

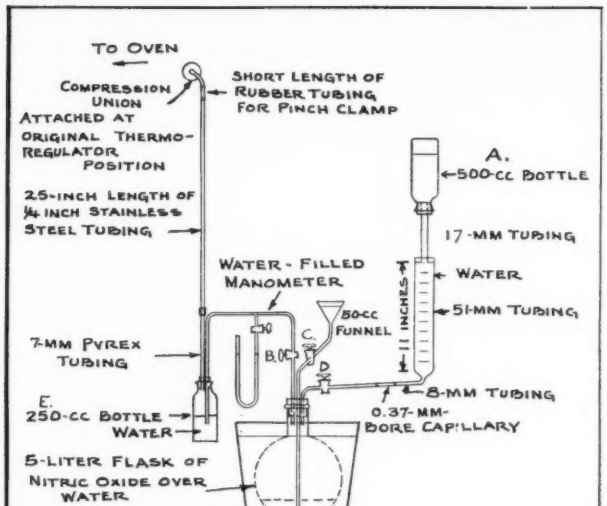

Figure 6
Equipment for Controlling Flow of Oxides of Nitrogen.

Constant Leveling Device for Entering Oxides of Nitrogen into the Oven

A diagram of the apparatus developed for entering oxides of nitrogen into the oven is given in Figure 6.

The essential features of the constant level device were a 12-inch calibrated cylinder, at the lower end of which was a short piece of tubing bent at right angles to the cylinder, a piece of capillary with a bore of 0.37 mm., stopcock D which connected with the flask of gas, and bottle A (of 250 to 500-c.c. capacity) fitted with a rubber stopper, and a short length of 17-mm. glass tubing. Water constituted the liquid used for displacing the gas. Its flow was kept at a constant rate by maintaining a constant level of water in the cylinder. This was achieved by inverting the water-filled bottle and tube so that the latter projected just to the desired level of liquid. It was found most convenient in these experiments to keep the level of the water one-eighth inch above the top calibration of the cylinder, and to vary the rate of flow when desired by changing the length of capillary used. Changing the height of the liquid column, however, would have provided another means of accomplishing the same purpose. The minimum head of liquid, or height of the liquid column below the lowest calibration was 4¼ inches. The distance from the bottom to the top calibration was 11 inches.

After the five-liter flask was filled with gas, as previously described, it was fastened accurately in a galvanized bucket which permitted it to be surrounded with water. To offset fluctuations in room temperature and subsequent expansion or contraction of the gas during a test, the water surrounding the flask was maintained at a constant temperature throughout any series of experiments.

It may be noted that the various stopcocks connected directly to the flask of gas have been given the same designations in the diagram under consideration as were used in Figure 5 illustrating the equipment for generating the gas. To stopcock C there was attached a short length of rubber tubing and a funnel through which water could be entered directly into the flask for the purpose of building up the

pressure of the gas after drops had occurred as a result of absorption of small amounts of gas by the water. Stopcock B was connected to a length of glass tubing which extended just below the surface of the water in bottle E. Another tube leading from the top of bottle E connected with the flange connection at the brass pipe leading into the oven.

The functions of bottle E were:

1. To serve as a trap to prevent air from the conditioning apparatus (which was under slight pressure) from getting into the flask of gas, and
2. to provide a means for determining when the gas started to flow, as well as for judging the uniformity of the flow of gas.

A short length of rubber tubing inserted close to the flange permitted shutting off of the gas with a pinch clamp at this point between test runs.

A water-filled manometer inserted by means of a tee between the gas flask and bottle E was used to measure the pressure of the gas.

In order to prevent leaks, all tubing connections (glass-to-glass) were wired and shellacked.

Equipment and Method for Collecting Sulfur Dioxide and for Entering it into the Oven

After a few preliminary experiments, mercury was selected as the displacing agent for sulfur dioxide because of the high solubility of the gas in other liquids. It, therefore, was deemed most practical to collect the gas in small bottles, to be replaced after one or two test runs in order to avoid the necessity for obtaining large quantities of mercury.

Wash bottles of 250-c.c. capacity were fitted with two-hole rubber stoppers which held two-inch lengths of glass tubing. Short pieces of rubber tubing were fitted over the ends of the glass tubes to permit closing them with pinch clamps. The bottles were filled with Merusol (mineral oil), which then was displaced with sulfur dioxide from a tank source. The gas was collected at a pressure somewhat greater than atmospheric, in order to offset the absorption which was found to lower the pressure to considerably less than that of the atmosphere, after one day only. Immediately after collecting the bottles of gas, all potential leaks including the edge of the stoppers were shellacked.

A diagram of the apparatus developed for entering sulfur dioxide into the oven is given in Figure 7.

The main features of this apparatus were similar in principle to those of the equipment for entering oxides of nitrogen into the gas-fading chamber. A 50-c.c. funnel with its tube bent to a 90° angle was used for the constant level reservoir. A 125-c.c. bottle, which could be refilled as needed, was the source of the mercury for maintaining the constant level. A capillary of 0.37-mm. bore was used, its length depending on the rate of flow desired. It was attached to a stopcock mounted in a tube of 2.0-mm. bore, which was connected to the bottle of gas. The exit tube from the gas bottle led to a Merusol-filled manometer, and finally to the previously described flange connection with the oven. Because of the fact that the bottle of gas was replaced after one to two test runs, no trap bottle was used.

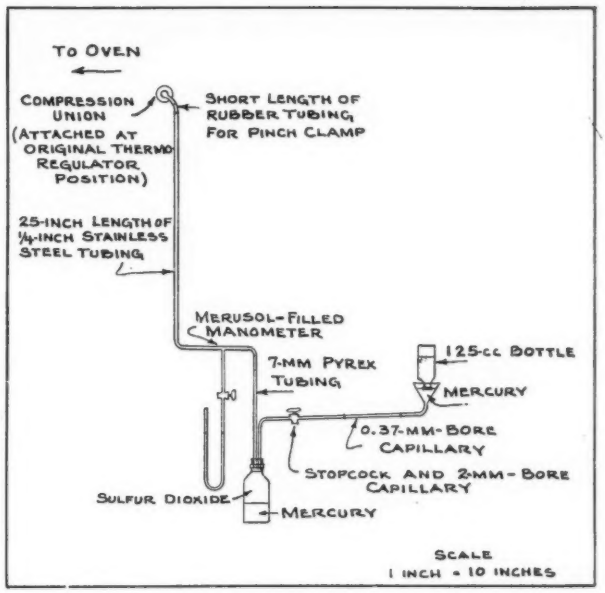

Figure 7
Equipment for Controlling Flow of Sulfur Dioxide.

Although sufficiently workable to contribute to the study, this set-up presented the following undesirable features in use:

1. Even after the sulfur dioxide was collected under pressure, it frequently was absorbed to the extent of 25 per cent over a period of three days. This made it necessary to collect only a few bottles of gas at a time and to use it within two days, and also to spend an undesirable amount of time to build up the pressure of the gas before starting each test run.
2. The mercury became contaminated with the oil clinging to the sides of the bottle, and, therefore, had to be filtered after each run.
3. With the use of the length of capillary necessary for the lowest rate of flow desired (approximately 50 c.c. of gas per hour at room conditions) the rate of flow of the mercury changed from time to time. Apparently small amounts of oil in the mercury or dirt which became lodged in the capillary changed the required head for the given rate of flow. This necessitated constant attention on the part of the operator to the number of drops of mercury per minute and called for frequent adjusting of the height of the funnel.

Equipment and Method for Entering A Combination of Gases into the Oven

Both oxides of nitrogen and oxides of sulfur could be entered into the oven by using in combination both sets of apparatus described above. The only adjustment required was the use of a tee, which was inserted at the lower end of the tube connecting with the oven and which connected with each type of gas.

Composite Description of Modifying Apparatus

A photograph of the gas-fading chamber with complete accessory equipment for controlling the rate of air flow, for conditioning the air, and for entering oxides of nitrogen into the apparatus is given in Figure 8. This photograph combines the various parts of the equipment diagrammed in Figures 3, 4, and 6.

When sulfur dioxide was used, the gas-fading apparatus shown in Figure 7 was substituted for that illustrated herewith, and was connected at the same point.

Figure 9 is a photograph of the complete equipment as set up for using a combination of oxides of nitrogen and oxides of sulfur. It represents a combination of Figures 3, 4, 6, and 7.

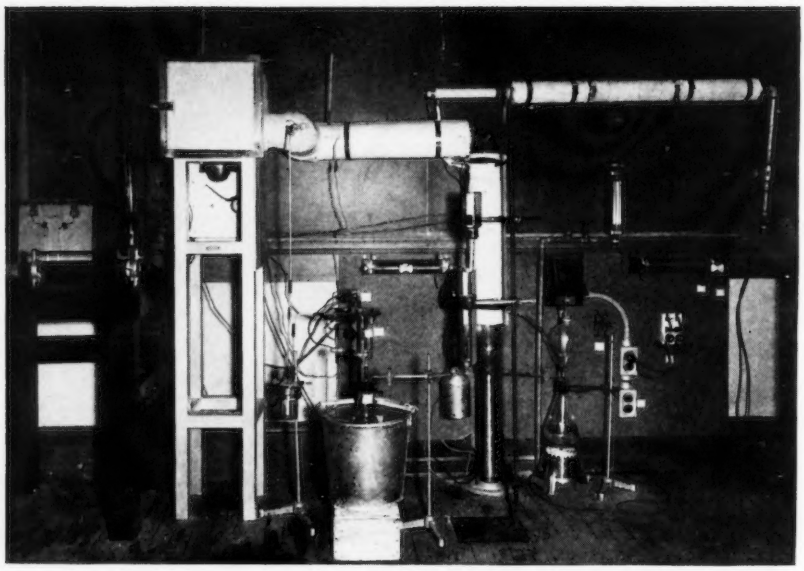

Figure 8
Gas-Fading Chamber with Air-Conditioning Apparatus and Equipment for Controlling Flow of Oxides of Nitrogen.

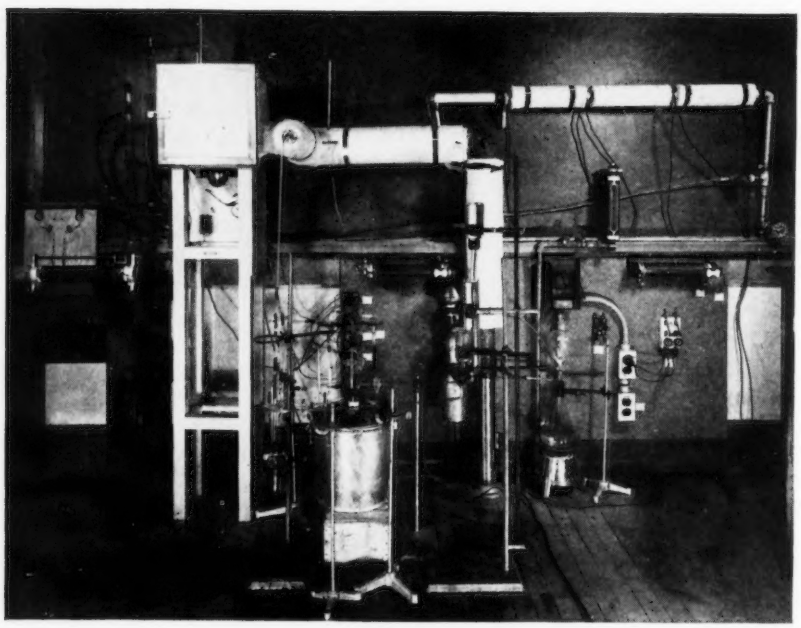

Figure 9
Gas-Fading Chamber with Air-Conditioning Apparatus and Equipment for Controlling Flow of a Combination of Gases.

Summary

Equipment especially designed for controlling the conditions believed to affect the nature and rate of gas fading in textile fabrics was tested and the necessary modifications made.

The plan to adjust the temperature and humidity of burnt gas fumes was found unsatisfactory and it was found necessary to enter chemically generated gas into a stream of conditioned air.

The supplementary equipment finally adopted for use with the gas-fading chamber included provision of means of controlling the rate of air flow, the oven temperature and humidity, and the rate of flow of the various destructive gases.

Further details of operation and data obtained with the use of this controllable equipment will be presented in future reports.

Acknowledgments

Sincere appreciation is expressed:

To Dr. Harold S. Ray for fundamental advice in the design and operation of the gas fading equipment;

To Mr. Charles W. Brouse and Mr. Francis J. Malloy for construction of special equipment;

To Dr. Pauline Beery Mack, Director, as well as to staff members of the Ellen H. Richards Institute for advice and assistance in the conduct of the study; and

To The American Viscose Corporation, under whose sponsorship this investigation was conducted at the Ellen H. Richards Institute, The Pennsylvania State College.

This is the fourth in a series of reports on methodology studies related to gas fading, from the laboratories of the Ellen H. Richards Institute, sponsored by the American Viscose Corporation.

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—♦ ♦—

• Report on Imports

The United States Tariff Commission recently issued a report on United States imports of coal-tar products entered in 1947 under paragraphs 27 and 28 of the Tariff Act of 1930.

In 1947 imports under paragraph 27 of all coal-tar intermediates totaled 2.6 million pounds, valued a $530,133. Corresponding imports in 1946 totaled 3.1 million pounds, valued at $437,169.

Imports under paragraph 28 of finished coal-tar products in 1947 amounted to 1.4 million pounds valued at $2,292,000 compared with imports in 1946 of 2.0 million pounds valued at $2,649,000. In 1947 as in previous years dyes constituted the most important group in imports of finished coal-tar products. They were imported almost entirely from Switzerland. Imports of dyes were less in 1947 than in 1946, but still accounted for about 88 percent of the finished coal-tar imports under paragraph 28.

Copies of the report may be obtained by writing to the United States Tariff Commission, Washington 25, D. C.

Proceedings of the
American Association of Textile Chemists and Colorists

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to Attend the Augusta Convention

from the Chairmen of the Southern Sections

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As co-sponsors of the 1948 National Convention, the Southeastern Section takes pride and pleasure in inviting members, guests and friends of the AATCC to Augusta, Georgia.

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The members of the Piedmont Section welcome the opportunity of joining with the other two Southern sections in sponsoring this National Convention and hope we have the opportunity of greeting you in Augusta.

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Our Committees are hard at work and are doing their utmost to make this a memorable event. We are pleased to have the opportunity of sponsoring this meeting and hope that you will enjoy yourself and want to return.

Raymond B. Seymour

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27th Annual Meeting and Convention

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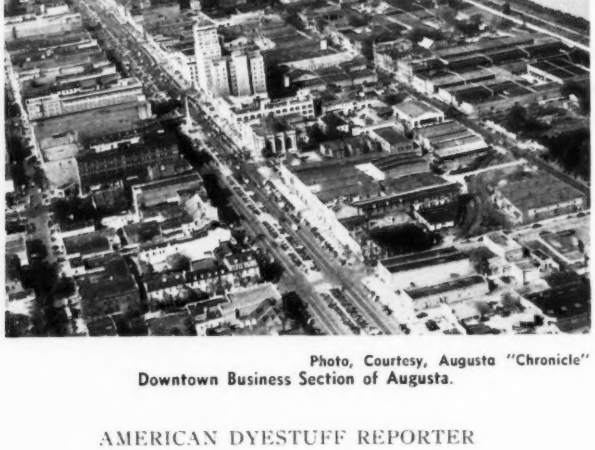

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The Dyeing of Woolen Fibers and Fabrics with Vat Colors*

G. T. HUG

Technical Laboratory, E. I. du Pont de Nemours & Co., Inc.

SINCE the turn of the century, when vat dyes other than indigo were introduced, it has been the aim of many investigators to devise suitable methods of applying these colors of exceptional fastness to wool. The damage to the wool fiber caused by the required methods of application has always prevented their extensive use, but we find numerous processes described in the literature, as well as in patents, which give detailed instructions for dyeing all forms of wool with vat colors. One conclusion, based on these references, is that it is essential to employ dyebaths of relatively mild alkalinity, and at the same time to use suitable protective agents in order to avoid or at least minimize wool damage.

In most cases we find recommendations for the use of dyebaths containing ammonia, hydrosulfite and animal glue *(1)*. Other authors advocate the reduction of alkalinity by the addition of various acids or acid forming salts *(2)*. Still others recommend the use of bases which are claimed to damage wool to a lesser extent than caustic soda, for example trisodium phosphate *(3)* as well as several of the organic bases *(4)*. One of the most recent methods to be tried is a modification of the vat acid or Kupensaure process. Here the acidified leuco compound is applied to the wool and the color finally fixed in the fiber by a re-reduction step under relatively mild alkaline conditions.

Many of these processes unquestionably have some merit, and it can be assumed that most of them have been tried out at one time or another on a mill scale, but it appears that the final results obtained have never been completely satisfactory since they have not had general acceptance. In checking some of these methods it was found that many of the anthraquinone type of vat dyes have little affinity for wool, producing relatively weak shades, and in quite a few instances the fastness properties were found inferior to what had been expected.

The early literature describing the dyeing of wool with vat colors is quite interesting. By early we refer to the "Twenties." We find the following said about it: "Anthraquinone vat dyes are unsuitable for wool due to the amounts of caustic soda required to put them into solution *(5)*. The dye described under Colour Index Number 1106, for example, requires a large amount of caustic soda and a dyeing temperature of 120-140° F. Its reduced leuco compound is not stable at low temperatures nor does it have much affinity for wool." *(6)* We have found, incidentally, that this particular color is a good one for wool! Even at that time it was recognized that for proper dyeing the leuco must have affinity for the wool fiber *(6)* and that when the dyebath is partially neutralized or reduced to a lower pH it is largely a question of precipitation on the fiber rather than of dyeing *(7)*. As stated before, while weakly alkaline dyebaths have been widely recommended *(3)* it has been recognized that under such conditions the affinity of vat colors for wool is lower than that for cotton *(8)*.

It becomes apparent that most workers on this problem followed cotton methods for dyeing wool, particularly in respect to dyeing temperatures, and that they attempted to modify the alkalinity of the dyebath and use protective agents to prevent damage to the animal fiber. At this low degree of alkalinity the true soluble leuco compound of the vat dye was no longer maintained in many cases, and affinity of the dye for wool was reduced, with the result that dyeings of relatively poor tinctorial value were obtained. A further reduction in color value was caused by the addition of wool protective agents to the dyebath, which appeared to be necessary at the dyeing temperatures of 120 to 140° F. generally used.

Only in very recent years has any serious attempt been made to increase the caustic soda concentration. In the soda ash process *(9)* it was demonstrated that wool could withstand relatively large concentrations of caustic soda under controlled conditions of temperature providing the time of contact was very short. Later recommendations for wool and mixed fiber fabrics containing wool have increased the caustic soda concentrations to almost 0.5 ounce per gallon, using a wool protective agent and dyeing temperatures of 120° F. and over *(10)*.

We have gone a step further in that the caustic soda concentration has been increased still more, the use of wool protective agents has been eliminated and we are now able to dye woolen fibers and fabrics with vat colors using conventional dyehouse equipment. Tinctorially we are obtaining as much strength on wool as is normally expected on cotton. In a few cases the color value on wool is better, and only in two or three instances is it inferior. While we do not expect this to be *the* method we hope it will help to pave the way for further development and perfection of vat color application to wool.

Before describing the vat color dyeing method we wish to raise the question, "Why vat colors on wool, and what are their advantages?" First, we believe that vat colors are not required or suitable for every purpose. In women's dress goods, for example, acid and chrome colors are generally satisfactory. The light fastness is adequate for the life of the garment and the dyes employed are normally fast to dry cleaning. If a shrink resistant, washable wool comes into general usage, however, vat colors will assume increasing importance because bright, light-fast, wash-fast colors, a must for high style, are possible only with vat colors.

At the present time automotive fabrics probably head the list in importance where fastness to light is a primary requirement. Next come upholstery and drapery fabrics, carpetings, men's suitings, particularly in the summer weights, bathing suits and blankets where a high degree of light and wet fastness is desired. The demand for vat dyed stock or top for sports shirts and knitting yarns also should be encouraged by the development of washable, shrink resistant wool.

* Presented at meetings, Rhode Island Section, March 26, 1948 and New York Section, May 7, 1948.

Dyeing of Piece Goods

The problem of applying vat colors to wool can be divided into two parts, the dyeing of woven fabrics or piece goods, and the dyeing of loose fibers such as rawstock, top or even yarns. Let us first turn to the dyeing of piece goods, which while it may be of lesser interest from a commercial standpoint at the present time and still in an experimental stage, is much more interesting as a problem of development. It has been a generally accepted fact that piece goods would have to be dyed in open width form, for which elaborate and not readily available equipment is necessary; although it has been shown that indigo can be dyed in the rope form *(11)*.

Although further mill trials are desirable to confirm fully our results, we have found that it is possible to apply all types of vat colors to woolen fabrics in the rope form, using conventional piece dye kettles or reels under conditions of alkalinity not too far removed from those generally employed in cotton dyeing. Temperature is the controlling factor in the production of evenly dyed wool, undamaged by alkali; and time of dyeing insures good penetration and tinctorial strength.

As it has been established that wool will withstand considerable concentrations of caustic soda at sufficiently low temperatures without suffering any appreciable degree of degradation, we recommend starting the dyeing at 90° F. or below, and in no case should a temperature of 110° F. be exceeded. There are two methods which have given satisfactory results in large scale tests, the reduced and the pigment method.

In the reduced method, which may be preferred for reasons stated later on, the vat color is reduced, at the normal temperature generally recommended for dyeing cotton, in one tenth of the total dyebath volume with 3 ounces per gallon each of caustic soda and sodium hydrosulfite.

The wool to be dyed is first wet out on a reel machine or piece dye kettle with 0.125 to 0.5 oz./gal. of a suitable wetting agent such as "Alkanol" WXN. The formation of foam appears to be very beneficial in wool dyeing, which is contrary to cotton dyeing experience. To this bath, comprising nine tenths of the total dyeing volume, 0.25 to 0.5 oz./gal. of caustic soda and 0.7 to 1.0 oz./gal. of sodium hydrosulfite are added. After running for 5 to 10 minutes the reduced color is added slowly. You will note that the caustic soda concentration is now between 0.525 and 0.75 oz./gal., while that of the hydrosulfite is between 0.93 and 1.3 oz./gal., the larger amounts being used for darker shades. The pH of this bath is somewhat above 11.

VAT COLOR DYEING—PIECE GOODS

Reduction: in 1/10 of dyeing volume
3.0 oz./gal. NaOH
3.0 oz./gal. $Na_2S_2O_4$
15 minutes at 140°F.

Dyebath: 9/10 of total dyeing volume
0.125-0.5 oz./gal. "Alkanol" WXN
0.25-0.5 oz./gal. NaOH
0.7-1.0 oz./gal. $Na_2S_2O_4$
Run 5-10 minutes at 90°F.
Add reduced color—run 30 min. at 90°F.
Raise Temp. to 100°F.—run 10 minutes
Raise temp. to 110°F.—run 10 minutes
Add 2.0-6.0 oz./gal. NaCl—run 15-20 min.
Add 1.0-1.5 oz./gal. $NaHCO_3$—run 5 min.

Rinse—oxidize with 2% $NaBO_2$

Figure 1.—Vat Colors on Woolen Fabrics, Dyeing Method for Reel Machines.

VAT COLOR DYEING—RAWSTOCK AND TOPS

Reduction: in 1/10 of dyeing volume
3.0 oz./gal. NaOH
3.0 oz./gal. $Na_2S_2O_4$
15 minutes at 140° F.

Spring: 9/10 of total dyeing volume
0.125-0.25 oz./gal. "Alkanol" WXN
0.25-0.35 oz./gal. NaOH
0.25-0.5 oz./gal. $Na_2S_2O_4$
Circulate 5 minutes at 90° F.
Add reduced color—circulate 10-15 min.
Raise temp. to 110°F.—circulate 15-20 min.
Add 2.0-6.0 oz./gal. NaCl—circulate 20-30 min.

Drop bath

Light shades: add 0.5 oz./gal. $NaHCO_3$
Circulate 5 minutes—rinse—oxidize

Dark shades: rinse—add 0.5 oz./gal. $NaHCO_3$
Circulate 5 minutes—rinse—oxidize

Figure 2.—Vat Colors on Woolen Rawstock and Worsted Tops. Dyeing Method for Circulating Machines.

Dyeing is now carried out as follows:

Run 30 to 60 minutes at 90° F.

Raise temperature to 100° F., run 10 minutes.

Raise temperature to 110° F., run 10 minutes.

Add 2 to 6 oz./gal. common salt, run 10 to 20 minutes.

The longer dyeing period at 90° F. is used for heavier or more tightly woven fabrics to insure adequate penetration, and the quantities of common salt used are dependent upon the depth of shade being dyed.

When the temperature of 110° F. has been reached it is essential to check both the caustic soda and hydrosulfite concentrations. For this purpose Clayton Yellow paper, which should show a faint red under proper conditions of alkalinity and "Ponsol" Yellow G paper, which must turn green instantly if sufficient hydrosulfite is present, are satisfactory. Where a deficiency of either caustic or hydro is noted, appropriate additions should be made immediately to prevent the vat from falling out of solution with resultant pigmentation and formation of streaks on the goods. There is practically no oxidation of the vat dye on the wool as it passes over the reel, even in open kettles.

After the dyeing cycle is completed the caustic soda remaining in the bath is immediately neutralized to soda ash by the addition of sodium bicarbonate, whereupon the bath is dropped. The material is rinsed, oxidized with sodium perborate

and soaped. The addition of bicarbonate to the dyebath is an important step in the production of bright shades of good tinctorial value and will be discussed later.

The pigment method eliminates the need for reducing the vat color prior to dyeing. The pigment color is added to the dye kettle and the goods run for about ½ hour at 90° F. Caustic soda and hydrosulfite, 0.6 to 0.75 and 0.9 to 1.3 oz./gal., respectively, are added and the same dyeing cycle is followed as was described for the reduced method.

Manipulation is somewhat simplified in this instance, as reducing the color in a separate container can be dispensed with. It is limited, however, to the use of such colors which can be reduced at temperatures of 90° to 100° F. For example, such colors as "Ponsol" Red G2B Double Paste and "Sulfanthrene" Pink FF Paste, a thioindigoid type, are not suitable for application by the pigment method. On the other hand, the pigment method lends itself better to the dyeing of wool-viscose mixtures than does the reduced method, as improved penetration of the viscose component is obtained.

Dyeing of Loose Fibers

Very substantial quantities of worsted tops and smaller amounts of rawstock have been dyed with vat colors, and we prefer to use conventional circulating machines of either the closed or open type. The procedure followed is almost exactly the same as has been described for piece goods. The spring bath is first circulated through the wool at 90° F. followed by the addition of reduced color.

Total concentrations of caustic soda and sodium hydrosulfite range from 0.5 oz./gal. each for light shades, to 0.65 and 0.75 oz./gal. respectively for darker shades. The vat dye is again reduced in one tenth of the total dyeing volume, and 0.125 to 0.25 oz./gal. of "Alkanol" WXN is sufficient for wetting out the wool.

The dyeing cycle is as follows:

Circulate spring bath 5 to 10 minutes at 90° F.

Add reduced color, circulate 10 to 15 minutes.

Raise temperature to 110° F., circulate 15 minutes.

Add 2 to 6 oz./gal. common salt, circulate 20 to 30 minutes.

Drop bath.

Contrary to the procedure for piece goods dyeing, sodium bicarbonate is not added directly to the dyebath. The machine is first drained and filled again with water containing the necessary amount of sodium bicarbonate, through the expansion tank. After circulating for 5 to 10 minutes the material is rinsed, oxidized and acidified. For dark shades, however, it is best to rinse immediately after dyeing, then neutralize with sodium

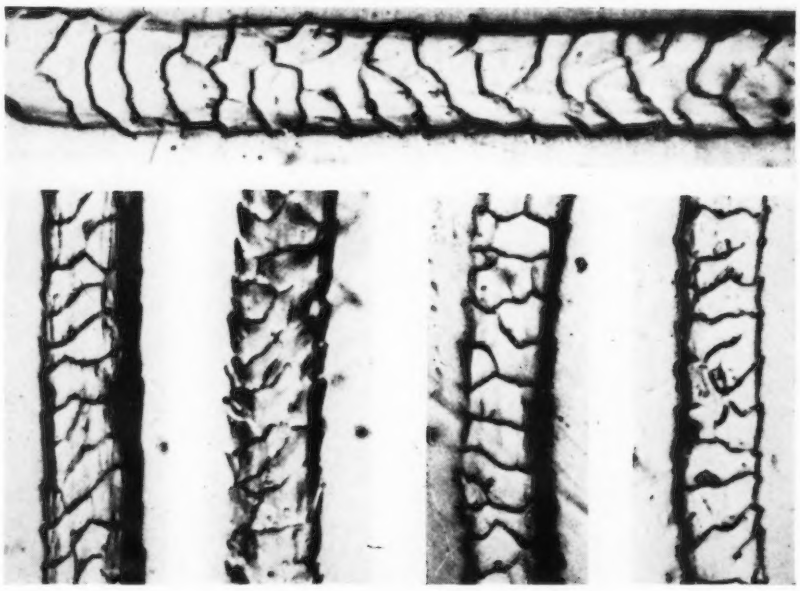


Figure 3.—Longitudinal Segments of Wool Fibers.
The horizontal fiber is undyed wool. The vertical fibers are, reading from left to right, dyed with vat, acid, chrome and "Chromacyl" colors.

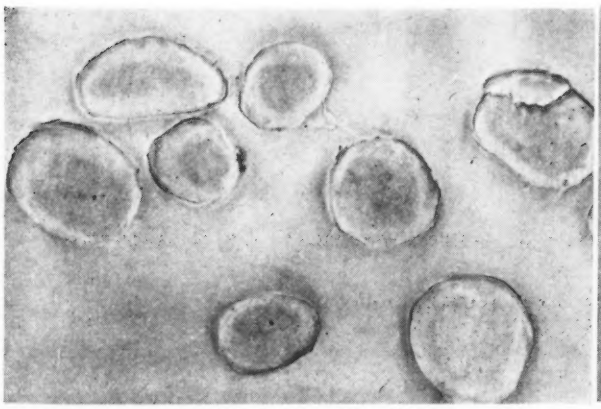


Figure 4.—Cross Section of Wool Dyed with 5 per cent "Ponsol" Red BN Double Paste (C.I. No. 1162).

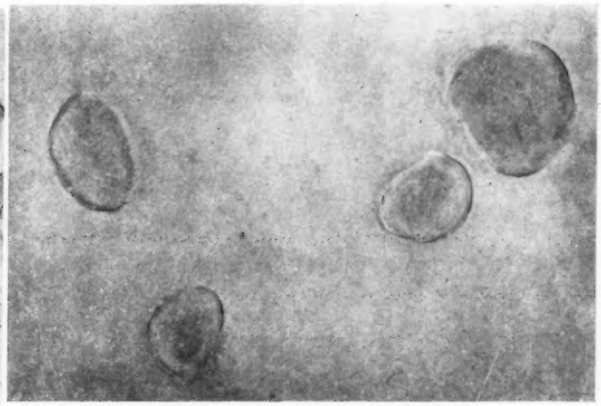


Figure 5.—Cross Section of Wool Dyed with
0.025% Du Pont Anthraquinone Blue SKY (C.I. No. 1054)
0.37 % "Pontacyl" Carmine 2G Conc. 150% (C.I. No. 31)
0.14 % Du Pont Tartrazine Conc. (C.I. No. 640)

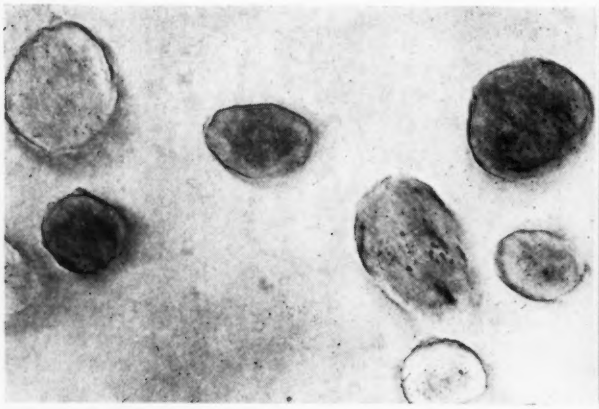


Figure 6.—Cross Section of Wool Dyed with
0.75% "Pontachrome" Fast Red 2RL (Pat.)
0.05% "Pontachrome" Flavine A Conc. 125% (C.I. No. 219)
Dyed Top Chrome

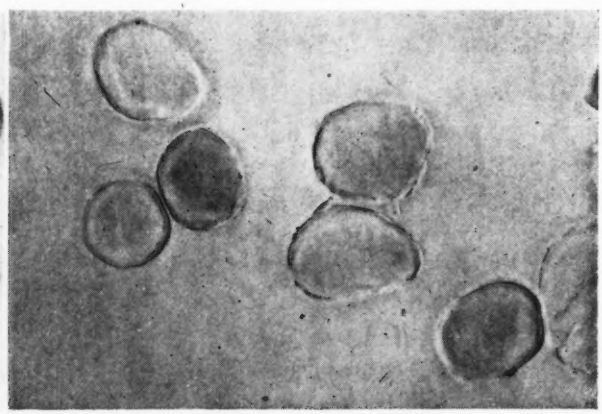


Figure 7.—Cross Section of Wool Dyed with
0.9 % "Chromacyl" Orange R (Pr. No. 146)
0.37% "Chromacyl" Pink B
0.01% "Chromacyl" Blue GG Conc. 200% (Pr. No. 144)

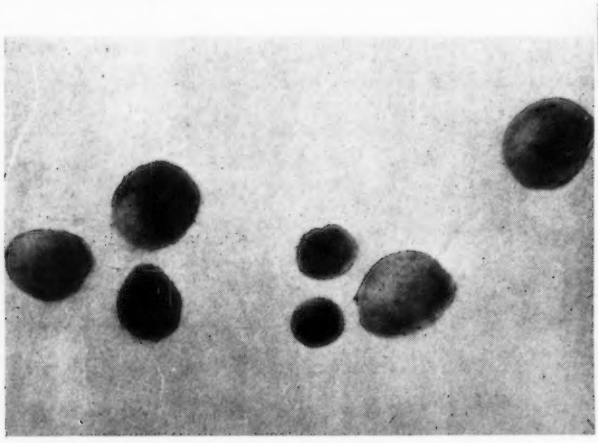


Figure 8.—Cross Section of Wool Dyed with
6.0% "Ponsol" Blue BF Double Paste (Pat.) (C.I. No. 1113)
3.5% "Ponsol" Direct Black 3G Double Paste (Pat.)
3.0% "Ponsol" Red BN Double Paste (C.I. No. 1162)

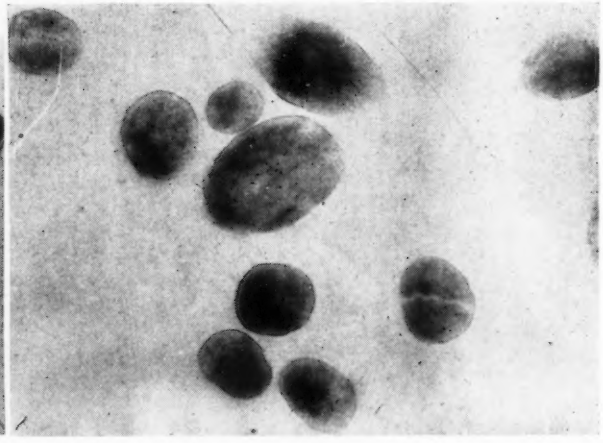


Figure 9.—Cross Section of Wool Dyed with
3.6% "Pontachrome" Fast Gray L
0.3% Du Pont Anthraquinone Blue SKY (Pat.) (C.I. No. 1088)
0.5% Du Pont Anthraquinone Violet 3R (C.I. No. 1080)
Dyed Chromate

bicarbonate. The resultant decrease in color value is more than offset by improved crocking fastness.

On rawstock and tops the pigment method has given satisfactory results in only a few instances, mainly for pale shades, and is not generally recommended.

In the dyeing of worsted tops it must be pointed out that the use of cotton covers or jackets should be avoided, particularly for pale shades. In some types of machines, covers are not necessary due to the low temperatures employed, but where needed we recommend they be made of wool, nylon or viscose. At the low dyeing temperatures used, vat colors generally have a much greater affinity for cotton than for wool, with the result that much of the available color will be absorbed by these cotton jackets.

TENSILE STRENGTH—ABRASION TESTS
Reel Dyed Flannel

	Warp	*Filling*	*Cycles*
I			
Original	68.0	48.0	..
Vat Dyed	66.5	46.0	..
Vat Dyed-Finished	59.0	36.5	2525
Acid Dyed-Finished	57.5	40.5	2000
II			
Vat Dyed	50.0	27.0	2475
Acid Dyed	44.0	27.0	2750
Premetallized Dyed	44.0	25.0	2425

Figure 10.—Tensile Strength and Abrasion Tests of Commercially Dyed Woolen Flannels.

The finishing treatment after dyeing is almost as important as the dyeing itself in preventing wool damage. Previous recommendations call for oxidation of vat dyed wool with acetic acid and peroxide, but this process may increase the harshness of the wool to a much greater extent than the caustic soda used in the dyeing cycle.

When acid oxidation or neutralization

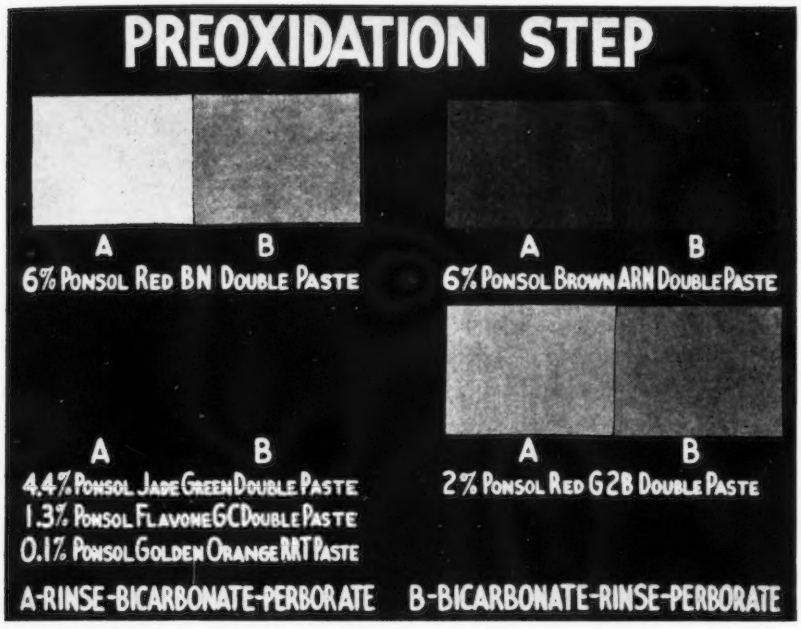


Figure 11.—Pre-oxidation Step. Effect of Addition of Sodium Bicarbonate to the Dyebath Prior to Rinsing and Oxidizing.

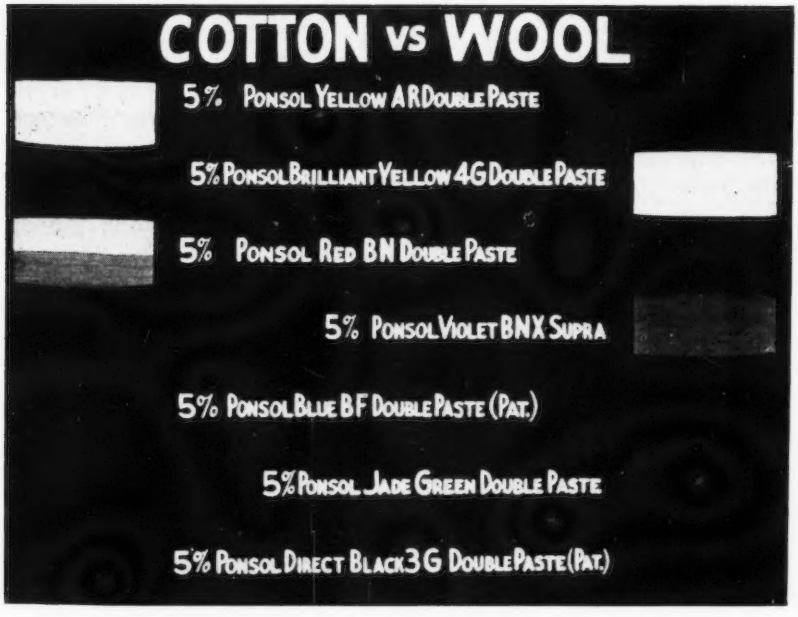

COTTON vs WOOL
5% PONSOL YELLOW AR DOUBLE PASTE
5% PONSOL BRILLIANT YELLOW 4G DOUBLE PASTE
5% PONSOL RED BN DOUBLE PASTE
5% PONSOL VIOLET BNX SUPRA
5% PONSOL BLUE BF DOUBLE PASTE (PAT.)
5% PONSOL JADE GREEN DOUBLE PASTE
5% PONSOL DIRECT BLACK 3G DOUBLE PASTE (PAT.)

Figure 13.—Comparative Tinctorial Strengths of Vat Dyes on Wool and on Cotton.

is employed and caustic alkali is still present in the wool, heat of reaction developed within the fiber is sufficiently severe to cause wool damage, with a resultant 25 per cent or more decrease in tensile strength besides imparting a feel of harshness. If caustic soda is neutralized with acetic acid at room temperature, approximately 13800 gram calories per gram mole are generated. But, when sodium bicarbonate is used only about 9100 gram calories are generated, with the added advantage that soda ash is formed, which is more readily washed out of wool than the stronger base. You all know that caustic soda is very hard to remove from your

TENSILE STRENGTH

Comparison of Oxidation Methods

	Relative Strength Per Cent
Control	100
Bicarbonate-perborate	98.5
Acetic acid-peroxide	76.5

Figure 12.—Tensile Strength of Woolen Yarns. Effect of Sodium Bicarbonate-Sodium Perborate Oxidation vs. Acetic Acid and Peroxide.

fingers with water alone and it is equally hard to wash out of a tightly packed mass of wool. Even after prolonged rinsing a considerable amount of caustic remains in the animal fiber. This heat of reaction is naturally confined within the fiber, is of short duration and is soon dissipated by the cold acidifying bath, but its action is sufficient to cause wool damage.

In addition, the tinctorial value of the dyeing also is influenced by the use of bicarbonate to reduce caustic alkalinity, particularly in the case of piece goods where washing is much more efficient than in rawstock or top dyeing machines. As long as caustic soda and hydrosulfite are present in the wool, the vat dye is, of course, still in its soluble leuco form and unlike on cotton, does not seem to have sufficient affinity to resist washing. The loss in tinctorial strength due to washing may amount to as much as 50 per cent. By adding bicarbonate to the bath, however, and forming soda ash within the fiber, the pH is reduced with a resulting reduction in solubility of the leuco compound. Then the wool can be washed without danger of removing a goodly portion of color.

This step is followed by normal oxidation with sodium perborate as has been previously described. After oxidation it is essential that the woolen material be well washed again. Piece goods should be soaped to remove any surface pigment, although this may not be necessary in the case of worsted top and rawstock where the usual wet finishing of the final fabrics will accomplish the same result.

Acidification as a final step is also important, since worsted tops are somewhat loftier after an acid treatment, and vat color dyed wool, particularly in paler shades, shows improved fastness to light if the material is neutral or even slightly on the acid side.

Selection of Dyestuffs

Perhaps the most important factor in promoting the general acceptance of vat colors for wool dyeing, is the selection of the dyestuffs. We have stated that all

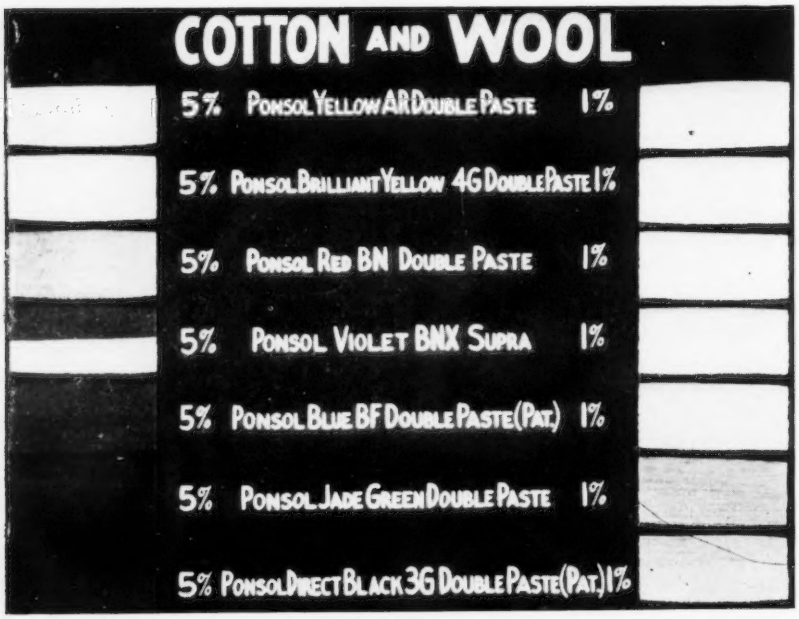

COTTON AND WOOL
5% PONSOL YELLOW AR DOUBLE PASTE 1%
5% PONSOL BRILLIANT YELLOW 4G DOUBLE PASTE 1%
5% PONSOL RED BN DOUBLE PASTE 1%
5% PONSOL VIOLET BNX SUPRA 1%
5% PONSOL BLUE BF DOUBLE PASTE (PAT.) 1%
5% PONSOL JADE GREEN DOUBLE PASTE 1%
5% PONSOL DIRECT BLACK 3G DOUBLE PASTE (PAT.) 1%

Figure 14. Comparative Tinctorial Strengths of Vat Dyes on Wool and Cotton Dyed in the Same Bath.

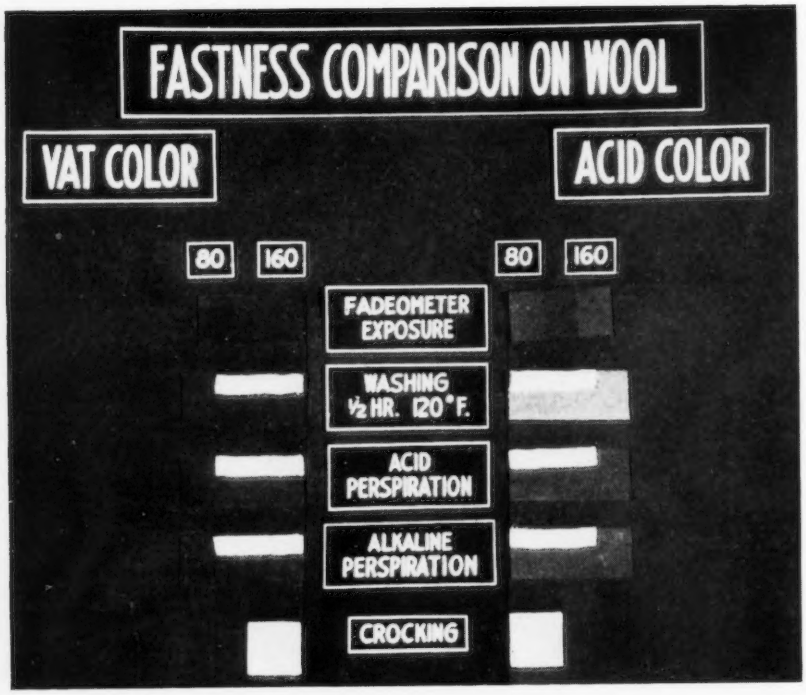


Figure 15.—Fastness Tests—Vat vs. Acid Colors
Vat Color Dyeing: 2.0% "Ponsol" Blue BF Dbl. Pst. (Pat.) (C.I. No. 1113)
1.0% "Ponsol" Jade Green Dbl. Pst. (Pat.) (C.I. No. 1101)
Acid Color Dyeing: 0.048% "Pontacyl" Light Yellow GX (C.I. No. 639)
0.912% Du Pont Anthraquinone Blue BN (C.I. No. 1054)

vat colors can be applied to wool by the methods described, but not all of them are by any means suitable for every purpose. Our knowledge of the subject is not complete as yet, but to date we find the following colors to be of greatest interest.

"Ponsol" Flavone GC Dbl. Paste (Pr. #9): is a greenish yellow of good tinctorial strength. Although it is only fair to light, it will have to be used as a shading component for bright green shades.

"Ponsol" Yellow AR Dbl. Paste (C.I. #1132): Very good light fastness and tinctorial strength, both being much superior than when dyed on cotton.

"Ponsol" Golden Orange RRT Paste (C.I. #1098): A bright orange of excellent tinctorial strength and light fastness.

"Ponsol" Brown ARD Dbl. Powder (C.I. #1151): Is useful as a base for medium to dark shades of brown. Unlike on cotton, the light fastness is only fair in pale shades.

"Ponsol" Brown RBT Paste: Not as red as Brown ARD. Not too fast to light in pale shades but a suitable base for darker browns.

"Ponsol" Red BN Double Paste (C.I. 1162): Is a very important shading color, very fast to light in pale shades.

"Ponsol" Red G2B Double Paste (Pr. #124): Is a bluish red of good tinctorial strength. Mainly recommended for use as a base color for red and maroon shades.

"Ponsol" Brilliant Violet 4RN Paste (C.I. #1104): Exhibits good tinctorial strength and light fastness but turns much redder under artificial light. Tends to dye somewhat unlevelly due to its rapid exhausting properties.

"Ponsol" Violet BNX Supra (C.I. #1163): Is very important as a shading component. Relatively weak tinctorially but of good light fastness.

"Ponsol" Violet AR Double Paste (C.I. #1135): Exhibits very good tinctorial properties and is useful for deep shades.

"Ponsol" Blue BF Double Paste (Pat.) (C.I. #1113): Is a very good blue for pale to medium shades, good tinctorial strength and good light fastness.

"Ponsol" Blue GD Double Paste (C.I. #1112): Is somewhat greener than Blue BF. Slightly better tinctorial value in deep shades than the former type.

"Ponsol" Blue 3G Paste (C.I. #1109): Is greener in shade than Blue GD.

"Ponsol" Navy Blue Dbl. Paste: Is an excellent base for dark navy shades although it goes redder under artificial light.

"Ponsol" Blue Green Y Dbl. Paste (Pat.): Exhibits very good tinctorial strength and is suitable as a base for dark green.

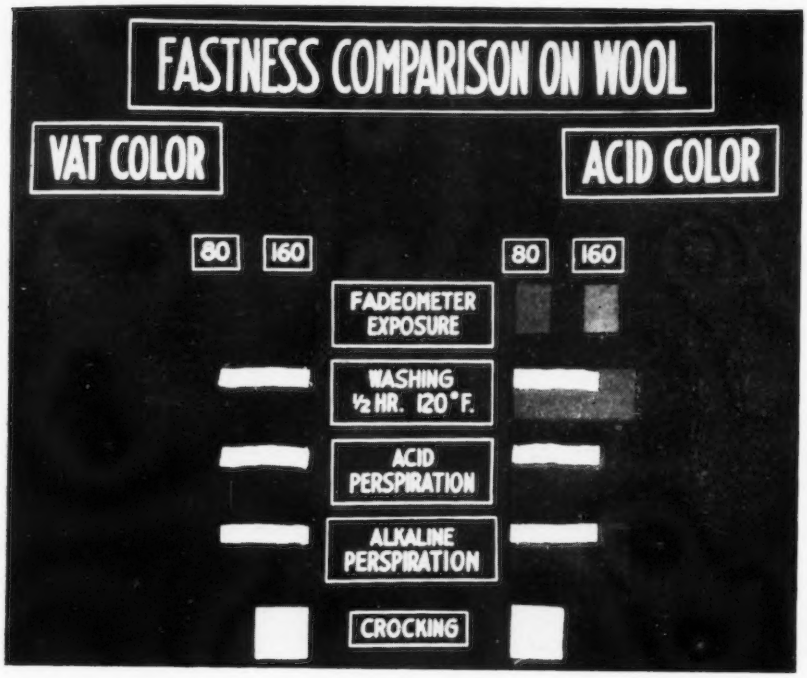


Figure 16.—Fastness Tests—Vat vs. Acid Colors
Vat Color Dyeing: 3.0% "Ponsol" Red G2B Double Paste (Pr. No. 124)
Acid Color Dyeing: 0.045% Du Pont Anthraquinone Blue BN (C.I. No. 1054)
0.85% "Pontacyl" Carmine 2G Conc. 150% (C.I. No. 31)
0.06% "Pontacyl" Light Yellow GX (C.I. No. 639)

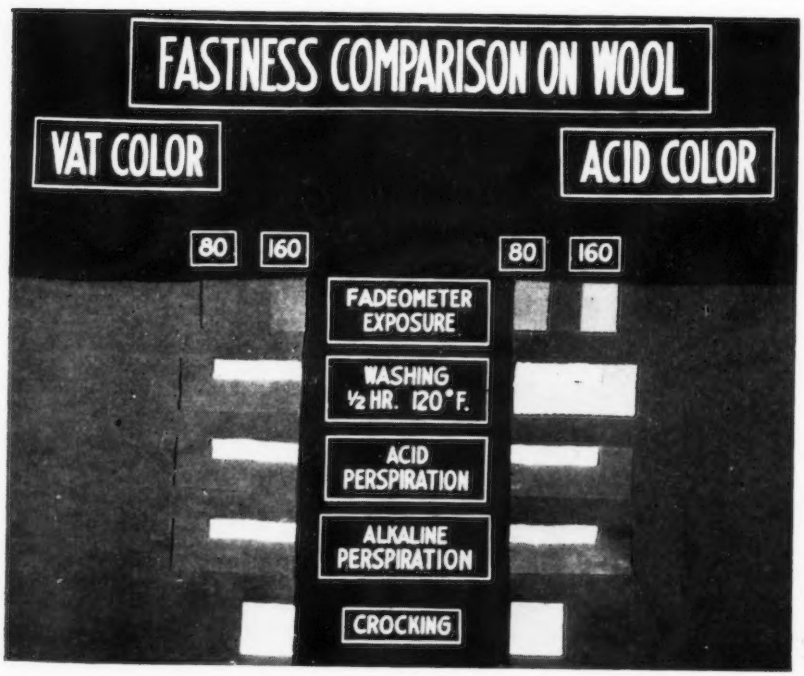


Figure 17.—Fastness Tests—Vat vs. Acid Colors
Vat Color Dyeing: 4.0% "Ponsol" Jade Green Dbl. Pst. (Pat.) (C.I. No. 1101)
Acid Color Dyeing: 0.28% Du Pont Anthraquinone Blue BN (C.I. No. 1054)
0.96% "Pontacyl" Green NV Ex. Conc. 200% (C.I. No. 735)

"Ponsol" Jade Green Dbl. Paste (Pat.) (C.I. #1101): Is the brightest green available. Exhibits very good tinctorial strength and is fast to light.

"Ponsol" Green 2BL Paste: Is a very good shading color with good light fastness.

"Ponsol" Olive GGL paste (Pat.) Is a duller shading color with fairly good light fastness.

"Ponsol" Direct Black 3G Dbl. Paste (Pat.): Is a neutral gray shade on wool, redder and with much better light fastness than when dyed on cotton. Is a very important shading color.

With reference to crocking and all wet fastness properties, the above mentioned colors are generally good.

For the production of tan or beige shades the use of a brown color as a base is to be avoided if maximum light fastness is desired. We recommend, for such shades, a mixture of Golden Orange RRT, Direct Black 3G, Red BN and Violet BNX Supra. Gray shades are best made with Direct Black 3G as a base, Blue BF or GD, shaded with Violet BNX, Red BN and sometimes Golden Orange RRT.

In conclusion we wish to point out that vat color dyeing of wool has progressed far beyond the laboratory stage. While much remains to be done in adapting the process to specific types of dyeing equipment and in the selection of dyestuffs to give ultimate fastness properties, large scale production has shown that duplication of results is obtained. Proper control of temperature, caustic soda concentration and time cycles, coupled with the proper selection of dyestuffs and treatment after dyeing will produce dyeings which surpass other existing colors in fastness properties and leave the wool in an undamaged, excellent condition for further processing.

. . . A number of slides were shown to illustrate more clearly some of the points discussed in the paper. These slides are represented by figures 1-21. . .

References

(1) Fox, Vat Dyestuffs and Vat Dyeing (p. 35).
(2) British Patents 209,569, 340,267.
German Patents 411,007, 438,244.
(3) Whattam, American Dyestuff Reporter, Vol. 25, p. 566.
(4) U. S. Patent 1,815,990.
British Patent 262,506.
German Patent 659,493.
French Patent 622,340.
(5) Cady, American Dyestuff Reporter, Vol. 16, p. 189.
(6) Myer, American Dyestuff Reporter, Vol. 19, p. 237.
(7) McDermott, American Dyestuff Reporter, Vol. 17, p. 322.
(8) Mayer, Monatschrift für Textil Industrie 50, 41 (1935).
(9) U. S. Patent 2,318,133.
(10) Clark, Amer. Dyestuff Reporter, Vol. 36, p. 269.
(11) U. S. Patent 2,420,729.

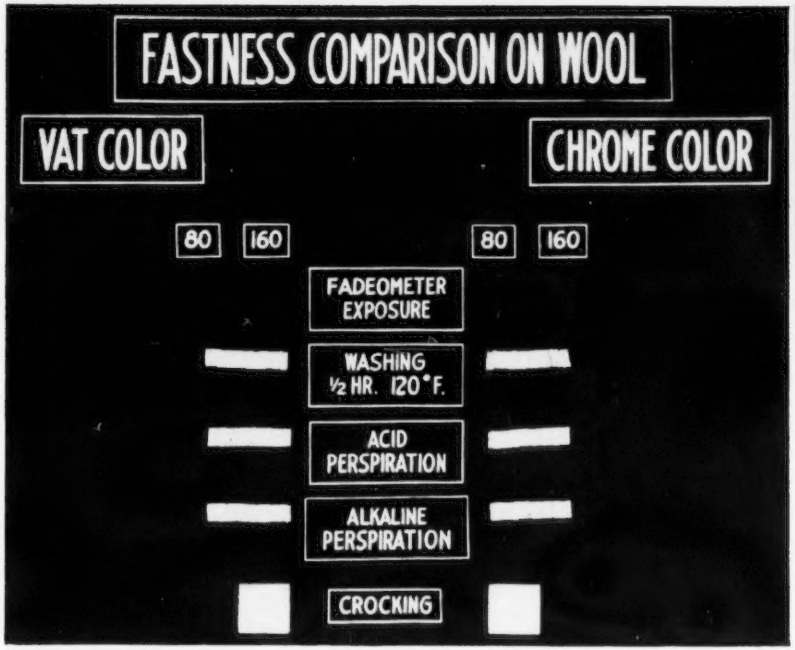


Figure 18.—Fastness Tests—Vat vs. Chrome Colors (Gray)
Vat Color Dyeing: 2.0% "Ponsol" Direct Black 3G Dbl. Pst. (Pat.)
0.5% "Ponsol" Blue BF Dbl. Pst. (Pat.) (C.I. No. 1113)
0.3% "Ponsol" Golden Orange RRT Pst. (C.I. No. 1098)
0.15% "Ponsol" Violet BNX Supra (C.I. No. 1163)
Chrome Color Dyeing: 0.176% "Pontachrome" Brown BGL
0.56% "Pontachrome" Fast Gray L
0.13% Du Pont Anthraquinone Rubine R Conc. (Pat.) (C.I. No. 1091)

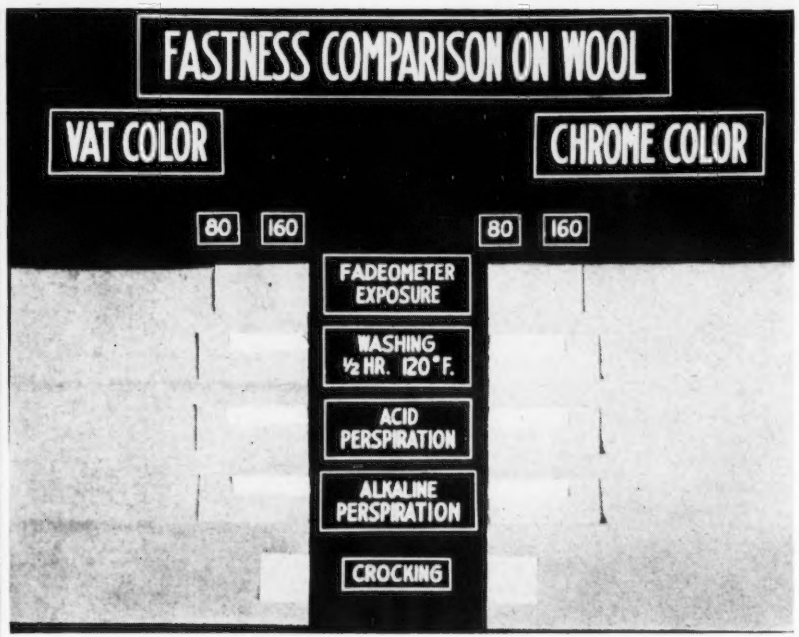


Figure 19.—Fastness Tests—Vat vs. Chrome Colors (Tan)
Vat Color Dyeing: 0.321% "Ponsol" Golden Orange RRTD Dbl. Pdr. (C.I. No. 1098)
0.55% "Ponsol" Direct Black 3GD Dbl. Pdr.
0.107% "Ponsol" Red BND Dbl. Pdr. (C.I. No. 1162)
0.701% "Ponsol" Violet BNX Supra (C.I. No. 1163)
Chrome Color Dyeing: 0.6% "Pontachrome" Brown BGL
0.05% "Pontachrome" Fast Gray L
0.025% Du Pont Anthraquinone Rubine R Conc. (C.I, No. 1091)

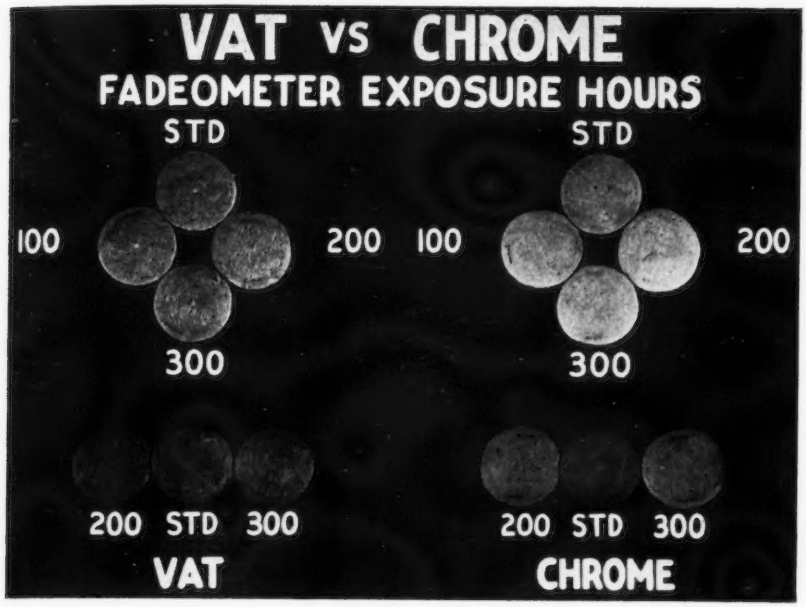


Figure 20.—Fade-Ometer Exposures—Vat vs. Chrome Colors

Top Row-Tan

Vat Color Dyeing: 0.321% "Ponsol" Golden Orange RRTD Dbl. Pdr. (C.I. No. 1098)
0.527% "Ponsol" Direct Black 3GD Dbl. Pdr.
0.125% "Ponsol" Red BND Dbl. Pdr. (C.I. No. 1162)
Chrome Color Dyeing: 0.4% "Pontachrome" Brown BGL
0.168% "Pontachrome" Fast Gray L
0.05% Du Pont Anthraquinone Rubine R Conc. (C.I. No. 1091)

Bottom Row—Gray

Vat Color Dyeing: 0.96% "Ponsol" Direct Black 3G Dbl. Pst. (Pat.)
0.71% "Ponsol" Blue GD Dbl. Pst. (C.I. No. 1112)
0.40% "Ponsol" Violet BNX Supra (C.I. No. 1163)
Chrome Color Dyeing: 0.4% "Pontachrome" Fast Gray L
0.075% Du Pont Anthraquinone Blue SKY (Pat.) (C.I. No. 1054)
0.09% Du Pont Anthraquinone Violet 3R (C.I. No. 1080)

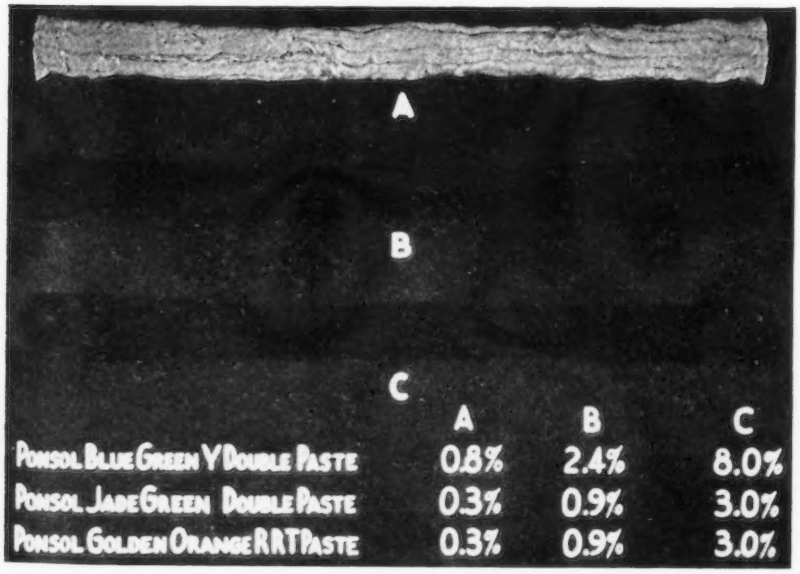

A
B
C
A B C
Ponsol Blue Green Y Double Paste 0.8% 2.4% 8.0%
Ponsol Jade Green Double Paste 0.3% 0.9% 3.0%
Ponsol Golden Orange RRT Paste 0.3% 0.9% 3.0%

Figure 21.—Build-up Properties of Vat Colors on Worsted Top. 1.4%, 4.2% and 14% Dyeings.

Discussion

(R. I. Section Meeting)

Q.—Have you done any work with Indigosol Dyes on worsted fabrics?

A.—No. All work to date has been with true vats.

Q.—What are the results obtained with the Vat-Acid Method?

A.—The fastness properties of the final shade were disappointing in some respects. The dyeing method is also more complicated than the present one.

Q.—How fast to perspiration are vat colors on wool?

A.—All tests made have indicated that vat colors are very fast. There is no staining and only a very slight shade change, in a few cases.

Q.—Is there any loss in weight of wool dyed with vats?

A.—There is a slight loss in weight.

Q.—What is the increase in alkali solubility of wool after vat dyeing?

A.—We have no available data.

Q.—Is the time cycle varied because of the high alkali concentration and would there be any advantage in cutting it down?

A.—The time cycle is closely controlled for duplication of shade. Any variances in time during dyeing would result in off shade material. Time has no great effect on the wool. The dyeing cycle is governed by the depth of shade. Light shades would require less time than dark shades.

Q.—Is the addition of salt necessary, and are larger or smaller quantities needed than when dyeing cottons?

A.—The salt is used only to promote better exhaustion. There is no data on quantities of salt or wool vs. cotton.

Q.—What is the comparable light fastness of vat vs. chrome when tested in the Fade-Ometer and in sunlight?

A.—Fade-Ometer tests indicate in most instances the superior light fastness of vats. There are, of course, vat colors which are only fair to light. These would be inferior to the better chrome colors. Results on sunlight are not yet available but will be as soon as tests are completed.

Q.—Can additions be made with vats on piece goods?

A.—No. However, shading can be done by using selected acid colors.

Correction

Re: "Nylon Dyeing" by Harry Clapham, page P299, May 3, 1948 issue

THE formula at the bottom of page P299 should read:

0.5—1.0% Acetic Acid 28% (based on the weight of the goods).

5-10% Tannic Acid (depending on depth of shade).

A Woolen Mill Dyehouse*

ERIC APPELTOFFT
Wanskuck Company

WE have all read and listened at various times to theories, new dyeing methods and research problems on wool. This information is always helpful, but we who have to manage a dyehouse and keep production rolling do not always have the time to treat our problems as a brain-child. Our theories and changes in dyeing methods are born of necessity and seldom can be treated as research problems.

Packing Kettle

In the dyeing of wool rawstock it is important that the wool be packed correctly in the dyekettle. This is especially the case when closed pressure kettles are used. Uneven packing can cause uneven dyeings, producing extreme light and dark sections. The temperature of the water to be used for packing the wool into the kettle depends on the type of stock used and varies from 120°-160° F. To this water is added a wetting agent or ammonia. Anywhere from 60-100 pounds are entered into the kettle at a time and allowed to be thoroughly wet out before the next layer is added. When all the wool has been entered into the dyekettle the water is allowed to circulate for about 10 minutes before cooling back to the starting temperature for dyeing. If necessary a penetrating agent is added along with the salt and dyestuff and allowed to circulate before the other chemicals are added.

During the dyeing procedure, if the proper selection of dyes is used, it is not necessary to cool back when making additions of acids, chrome or dyes for shading. All you have to do is to turn off the steam, dilute your additions, add slowly over a period of 5 minutes to the dyebath, then turn on the steam and keep on dyeing.

This is quite important in pressure dyeing machines, inasmuch as every time you have to cool back you must drop some of the dye liquor. That has a tendency to compress the wool, and the bottom layer in the dyekettle becomes very solidly packed, making it difficult for further additions to penetrate and causing much lighter dyeings on the bottom of the load than normally obtained. However, in the case of dyeing black, where the chroming is carried out in a fresh bath, this seldom happens; as the black dyestuffs chrome much more readily than other dyestuffs, and the slightest amount of chrome and acid penetrating is enough to chrome them over.

After dyeing the bath is dropped, and the wool given a rinsing to cool it. Often it is necessary to give the wool a prescour before dyeing, and here it is advisable to add enough chemicals at the start so that only one pre-scour is needed to clean the wool suitably for dyeing, as a second scour will only pack the wool that much harder and make it harder to penetrate.

Varied Affinity of Wool

They say that a man's disposition depends largely on what he eats. Well so far as the wool dyer is concerned, no more unpredictable creature than the sheep as regards the outcome of its eating habits exists. We know that the same sheep will produce wool with different affinities for dyestuffs and chemicals depending on whether they were grazing in Montana, Ohio or Texas. Sometimes I think, judging from dyeings on wool supposed to be all one lot, that the sheep with wool in that lot must have been dreaming some night of grazing in different pastures. Some years ago, for exhibition purposes, I obtained various pound samples of different grades of wool from all over the country. Samples of these were dyed in one bath with different dyestuff combinations and dye methods. The difference in shade between wool samples of the same grade was in many cases just as large as between those of low and high count. Because of this varied affinity of wool for dyestuffs, the dyer must know his dyes, using those which have the same dyeing affinity. For many reasons this is not always possible; but thanks to the chemical industry, there are many useful dyeing aids on the market today which help overcome many of the dyeing faults caused by the varied affinity of wool.

If time permits, a laboratory dyeing should be made every time a new wool lot is used, as the change in affinity for the dyestuff can be quite startling.

Some time ago, one of our domestic clips gave the dyers a headache, inasmuch as the dyeings obtained were peppered with little white specks. I believe I tried every well known penetration and wetting agent available. A few helped a little, but none were satisfactory. At the time, I thought I was the only one having this trouble, but inquiries showed that others were too. In fact, an article was published under the heading "Elimination of Airbells in Raw-stock Dyeing". With eagerness I read the article, only to find out that no known product had been found to eliminate this fault. Chrome bottom dyeing was the only method which we knew could correct this trouble, but this was unsatisfactory for our use. We found the solution by giving a pre-scour with ammonia, trisodium phosphate and a detergent, but had to go as high as 170° F.

A topchrome formula we had been using for some time suddenly went sour. The bottom part of the dyeing at a depth of about 4 inches remained red and would not chrome over. We could take htis unchromed portion, give it a chroming in a fresh bath, and obtain the true shade. This condition was overcome by adding at the start ½% Metachrome Mordant.

Selection of Dyeing Method

In regard to which dyeing method is most desirable for wool raw stock, chromate dyeing is best for simplicity and time saving. However, fastness properties, some shades, and above all present cost prohibit the wider use of this method; so the chrome topped method is most used.

In the dyeing of piece gods, the dye method depends on the type of cloth and fastness properties required.

For acid piece dyes, cloth can be taken directly from the carbonizer and wet out in the dyekettle, thereby utilizing the acid in the goods. Level dyeing acid dyes are used, and practically every shade can be produced with these dyes.

The acid in piece goods can also be

* Presented at meeting, Rhode Island Section, March 26, 1948.

utilized in dyeing the metallized acid colors. For some shades more acid has to be added. This type of dyestuffs produces dyeings almost equal in fastness to chrome dyeings.

The only difference in the dyeing of the two mentioned types, is that after the dyebath is brought to a boil in ¾ to 1 hour, the acid color dyeings are complete after boiling for one hour, while the metallized requires 1½ hours.

In the dyeing of piece goods according to the chrome topped method, the kettle is brought to a boil in one hour, boiled ½ hour, cooled back to 160°, second acid added, boiled for ½ hour, cooled back to 160°, chrome added, and boiled for one hour. Chromate dyeings are brought to the boil in one hour and boiled for 1½ hours. Silk whites are produced with chrome dyes which leave the silk clean. The dyeing procedure is practically the same as for chrome topped dyeings, with the exception that only acetic acid is used, and the chroming is carried out in a fresh bath.

Chrome bottom dyeings are produced by chroming the pieces first, then dyeing in a fresh bath. This method is quite lengthy, but in some cases produces better looking dyeings, as it covers up certain manufacturing defects.

Dyeing and Finishing Faults

The only way wrinkles in the cloth can be produced in the dyehouse is by running in too low a dye liquor, cooling back too fast, or crowding the kettle. If wrinkles do not occur, and you know that none of these facts exist in your dyeing operation, then they have been caused somewhere in the wet finishing. In this case it is advisable to have the pieces crabbed before dyeing.

Uneven dyeings can be caused by unclean goods, rolling of the pieces, uneven carbonizing or neutralizing if the goods are carbonized first, faulty fulling, dyestuff going on too hot at the start, or dyestuff adds going on in too hot a bath and not distributing evenly. Sometimes the dyestuff addition is too concentrated. A lot of dyeing trouble can be eliminated by having the upper part of the baffle board which forms the feed box a solid piece without holes. Then the dye gets a good mixing and does not hit the pieces on the surface, because it has to sink down, and enter the part of the kettle containing the pieces through the holes in the lower half of the partition.

The Hatfields and McCoys could have learned a lot about feuding if they had watched the old time dyers and finishers. We can thank the textile schools, our own association, the dyestuff and chemical corporations, that the dyer and finisher realize how much they have to understand each other's problems.

In the dyeing of raw stock the dyer has to know, beside the regular fastness properties required from the wearing standpoint, what the finisher is going to do with the cloth after it is woven. Will it be fulled for 1 hour or 4 hours? If it is to be a multicolored effect, then the dyes used must not only stand the loss in depth, but also be fast to staining. Whether it is to be carbonized at a high or low degree of acid, or decated afterwards, the knowledge of these facts determine his selection of dyes.

The finisher has just as many headaches as the dyer; his source of trouble too lies in the wool used. Today most mills use rope soapers, which help eliminate uneven fulling caused by uneven soaping of the goods going into the fulling mill. But sometimes pieces made from supposedly the same wool lot will shrink differently, dry out faster and create more heat than normally. In the washers soap and grease will not wash out readily. This, in piece dyeing, can cause the things I mentioned before. The pieces produced from stock dyeing, if not clean, will become blotchy after passing through the carbonizer. If the pieces are not neutralized well after carbonizing, the heat of the decater or presses will cause acid remaining in the goods to react and change the shade. Carbonizing and subsequent neutralizing of woolen cloth is an important factor in woolen goods manufactured. Not enough importance can be stressed on its control, as many faults can be caused by it and they are hard to trace.

If the pieces are to have high luster, they are steamed, then rolled and allowed to stand. The length of time they steam and stand helps regulate the luster. If they are to be piece dyed, the finisher must see that all the pieces have the same time treatment; otherwise the pieces which have stood the longest will dye much darker.

GUSTAVE HUG
Speaker

ERIC APPELTOFFT
Speaker

AMONG THOSE PRESENT AT MARCH MEETING OF RHODE ISLAND SECTION

RAYMOND JACOBY, Chairman, Rhode Island Section, and WILLIAM H. CADY, National Past President

EMORY RICHARDS
Secretary, R. I. Section

FRANK McCOOL

Shrinkage Control of Wool by Wet Chlorination*

LEONARD SHAPIRO
*Textile Research Director, Alrose Chemical Co.***

A. Introduction

WOOL is a fiber noted not only for its outstanding value as a textile material, but also for its unique and sometimes unpredictable behavior. Not the least of its idiosyncrasies is its property of felting and shrinking under certain conditions of physical, chemical and mechanical treatment to form a solid mass of firmly interlocked fibers. While this property is very useful for some purposes, it may frequently result in a garment becoming unusable in a single washing. Because of the growing quality consciousness of consumers, retailers and manufacturers, ever-increasing pressure is being exerted on the wool processors for production of shrink-resistant fabrics and garments.

In the search for an ideal shrinkage-control processes, many false starts have been made in the past, and much material sold as "shrinkproof" in other countries has not been fully satisfactory. Evidently misled by the apparent simplicity of the chemical reactions involved, processors have failed to employ proper chemical control or to make control tests to determine whether each lot of material is actually shrink-resistant. Reasonably accurate control of chemical concentrations, temperatures, and pH is essential if uniformity from one batch to another is desired. Standard laundering tests should be run on all batches processed, to determine the degree of control and to *predetermine* the finished fabric or garment dimensions.

The distinction between *felting shrinkage* and *relaxation shrinkage* is still not adequately appreciated by all manufacturers. Chemical treatments, in general, render the wool resistant to *felting shrinkage only.* If felt-resistant wool is knitted or woven under tension or stretched in drying, it is temporarily "set" in the stretched state. On immersing in water or scouring with very little agitation, some of the strains are relaxed, and the fabric comes in to its normal *relaxed* dimensions *without* any felting. In the case of many knitted garments, the fabric may be pulled out again with very little tension, but with woven fabrics, and some types of knit materials, relaxation shrinkage may be more serious. "London shrinking" is frequently employed to obtain partial relaxation of suiting materials. In other cases, steaming operations or mechanical preshrinkage are employed. Where preshrinkage cannot be employed, it may be necessary to oversize the garments slightly.

Now that satisfactory chemical shrinkage control methods are available, there is little doubt that the consumer will demand a minimum of both relaxation shrinkage and felting shrinkage.

One complication is the fact that wool fabrics do not always relax completely in the standard hot water or steaming treatments. When shrink-resistant yarns are employed, knit fabrics having a loose stitch construction will, even after relaxing, exhibit *consolidation shrinkage* on laundering (even though no felting occurs). By properly balancing the yarn number, stitches per inch and courses per inch, this consolidation shrinkage (as well as subsequent felting shrinkage) can be reduced to a minimum *(13).*

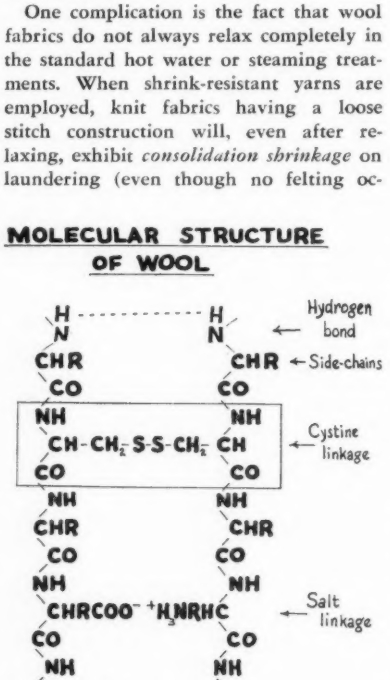


Fig. 1—Molecular Structure of Wool Keratin Illustrating Various Types of Cross-linkages.

B. Characteristics of the Wool Fiber

The wool fiber is a network of long polypeptide chains linked together by disulfide groupings, ionic salt linkages and hydrogen bonds. The long chains are studded with aliphatic side chains which comprise nearly half the total molecular structure.

In Fig. 1, the chemical structure of wool keratin has been greatly simplified to illustrate the various types of linkages and side-chains. Actually, the structure is extremely complicated, since at least 18 different amino acid residues are present in the peptide chains, and the various linkages join large numbers of chains into a three-dimensional network. The physical and chemical properties of the wool fiber are determined to some degree by the cross-linkages and the configuration of the side chains.

The remarkable elasticity and resiliency of wool is accounted for by the fact that the long molecular chains are not straight as shown in Fig. 1, but actually assume a folded and coiled configuration. When the fiber is stretched, the folded molecular chains (which cannot slip past each other because of the cross-linkages) straighten out, and when the tension is released, the molecules return to their coiled form again.

Physically, the wool fiber contains three structural components. Surrounding the fiber is the *cuticle*, only 2 or 3 microns thick and consisting of overlapping microscopic scales. The scales are similar in chemical structure to the remainder of the fiber, but have more frequent cross-linkages. For this reason they are tougher and more resistant to chemical attack than the keratin in the main portion of the fiber, which consists of fibrous *cortical cells.*

* Presented at meetings, Rhode Island Section, November 28, 1947 and Mid-West Section, February 28, 1948.

** Present position, Research Director, Synthron, Inc.

Many types of wool fibers also have a *central canal* containing pithy *medullary* cells.

Of extreme significance from the dyer's and finisher's standpoint is the tremendous *variation* in physical and chemical properties of wool fibers from various sources. Differences in breed of sheep, geographical locality, age of sheep, season of clipping, and location of hair on the fleece all exert a great influence on dyeing and felting properties. In a single wool fiber dyeing properties vary from root to tip, especially if the tip ends have been exposed excessively to sunlight and weather.

Wool processors are well aware of these many variations, and by blending wools with various properties, can control the working properties of the finished fabric.

C. Causes and Mechanism of Felting

The causes of felting have not been completely explained; however, it has been shown that the scale or cuticle layer principally is involved. The outer layers of molecules in the scales are believed to be oriented to an angle to the surface *(1)*. This causes a difference in friction between the root and the tip directions known as the "Differential Friction Effect" (DFE). On milling or laundering, owing to the elasticity and curling tendencies of the fiber and the differential friction effect, the wool fibers work their way rootward and become so firmly intertwined and matted that they cannot be separated *(2)*.

This unique characteristic of wool, known as felting, is accompanied by marked increase in thickness and shrinkage of the fabric which is known as felting, fulling, or milling shrinkage.

Felting shrinkage is used to great advantage in manufacturing clothing, hats, flannels, blankets, paper-maker's and industrial felts, etc. On the other hand, the felting shrinkage of wool is its greatest disadvantage in wearing apparel which is to be washed or laundered.

The amount of mechanical action, the pH and temperature of the bath, the type or fineness of wool, and the yarn and fabric structure all affect the ease with which felting takes place. Finer wools felt very readily, while coarse wools felt only slightly, or in some cases not at all.

The number and the angle of the contacts between fibers and the relative freedom of motion seem to be especially important. Thus *worsted* yarns and fabrics, which are made from combed tops, felt much more readily than *woolens* in which the fibers are not so parallel. Likewise slubbing is felted more readily than yarn, since in slubbing the fibers are not only more parallel but also more free to move in contact with each other. Yarn in skeins felts more readily than the same yarn knitted or woven into a fabric, since the yarns then cross at an angle and the fiber contacts are greatly reduced. For the same reason plied yarns felt less readily than singles, and tightly-woven or tightly- knitted materials felt less readily than looser constructions in which the fibers are less constrained.

The effect of type of stitch on the angle of yarn contact in knit goods, and hence on ease of felting, is illustrated by the fact that a plain stitch material will often felt badly on washing, while a rib stitch material from the same yarn will show very little felting. A considerable proportion of other non-felting staple fibers such as cotton, rayon, acetate or nylon, prevents felting by reducing the number of contacts between wool fibers.

D. Methods of Reducing Felting Shrinkage

Any treatment which reduces the differential friction effect of the scale surfaces, or which modifies the elasticity or elastic recovery of the fibers, is more or less effective in overcoming felting. Reduction of the differential friction effect has been accomplished by *(3)* by:

(1) Modifying the surface layers of the scales by chemical alteration in the molecules.

(2) Removal of scales or of surface layer of scales by chemical action or mechanical abrasion.

(3) Masking the surface of the scales with resins or with insoluble compounds.

While resin treatments have been successfully used on a limited range of fabrics, in the author's opinion, chemical treatments, when properly controlled, have less effect on hand and wool character, and are more versatile in application, especially on yarn, top, and knit gods.

Various reagents including alkalies, chlorine, oxidizing agents, reducing agents and enzymes have been employed to produce the desired effect. All of these agents appear to function by attacking the cystine grouping at the disulfide linkage. This breaks the primary valence forces between the molecular chains and permits some rearrangement to take place. In some cases, these linkages are later restored or replaced by other type linkages.

The conditions which cause breakage of the disulfide linkage can, under some conditions, become severe enough to cause breakdown in the main molecular chains. Hence the principal problem in the chemical method of treatment is to restrict the action to the disulfide linkages only, or to limit the action to the surface scale layers. Since the scales are more resistant to chemical attack than the interior of the fiber, the problem is by no means simple, and results tend to vary on different types of wool, depending on their scale and surface structure.

Of the vast number of chemical methods which have been proposed, only the alcoholic potash, dry chlorination, wet chlorination, enzyme and sulfuryl chloride methods have attained some degree of commercial usage. In several of these the degree of chemical action is regulated by carefully controlling the amount of water present during the reaction. Since this requires special equipment, commercial practice has swung again in favor of wet chlorination processes.

E. Wet Chlorination

The history of wet chlorination processes goes back to 1839 when John Mercer reported an increase in affinity of chlorinated wool for dyestuffs, a phenomenon used to advantage in printing on wool.

Shrinkage control of wool by wet chlorination was practiced in England before 1900, but until recently, even with the best process controls available, the methods were unreliable and results were difficult to reproduce. Overchlorination frequently resulted in degradation and loss of wool, and tensile strength and resiliency were impaired. Such wool retained excessive amounts of water, felt slimy when wet, and dyed unevenly.

During the last decade, however, the wet chlorination of wool has been re-investigated in the light of our greatly increased knowledge of the chemical and physical structure of wool, and of the chemistry of chlorination. Careful control of pH, amount of chlorine and uniformity of reaction have been found the essential factors in successful chlorination.

Some doubt still exists concerning the course of the chemical reaction. One theory is that the chlorine oxidizes the disulfide linkages of the wool molecule, forming among other products, cysteic acid groups:

$$\text{—CH}_2\text{—S—S—CH}_2\text{—} \xrightarrow{\text{Cl}_2} \text{--CH}_2\text{SO}_3\text{H} + \text{HO}_3\text{SCH}_2\text{--}$$

This is accompanied by a disarrangement of the surface layers of the cuticle and a reduction in the differential friction effect, rendering the fiber resistant to felting.

It is interesting to note that chlorination of wool is in final analysis an *oxidation* reaction rather than a true chlorina-

A—No Further Treatment

B—Chlorinated as in Figure 4 (Special Pretreatment Method)

C—Peroxide Bleached

Figure 2—Scale Structure of Wool—780X; 64s Australian Wool— Carbonized and Scoured. Note that the Effect of Chlorination on Scale Structure is Less than That Caused by an Ordinary Peroxide Bleach.

tion. No appreciable amount of organically bound chlorine has been reported in chlorinated wools, although it is likely that intermediate compounds are formed in which chlorine is attached to nitrogen or sulfur atoms.

The percentage of chlorine required for stabilization varies for different types of wool and should first be determined by laboratory trials. Coarser qualities of wool require lower percentages, and the finer grades higher percentages of chlorine with more careful handling. Different chlorinating processes require different quantities of chlorine for effective treatment, and the method of spinning, whether singles or plied yarn, twist, stretch, prior treatment and many other factors have a profound influence on the results. The presence of re-worked or bleached wool, which may have been damaged in prior treatment, can become a disturbing factor.

F. Chlorinating Bath

Chlorine is generally employed in the form of sodium hypochlorite or calcium hypochlorite. The former is known commercially as soda bleach, and is formed by bubbling gaseous chlorine into cold caustic soda solution:

$$Cl_2 + 2NaOH \rightleftharpoons NaOCl + NaCl + H_2O$$

Soda bleach is sold in concentrations of 10-15% "available chlorine" and contains 2-4% free caustic soda. It tends to lose chlorine at a rate of about 0.075% Cl per day at rom temperature, hence should be titrated to determine its exact strength before use.

Calcium hypochlorite, which can be used in many applications, is available as a powder containing about 70% available chlorine. This compound is much more stable to storage than soda bleach.

The chemistry of chlorination of wool is complicated by the fact that a number of types of reactions may occur. In alkaline solution (pH 9 or more) the chlorine occurs in the hypochlorite ion (—OCl^-). At pH of about 5.5, it is all in the form of hypochlorous acid (HOCl) which acts as an oxidizing agent:

$$HOCl \longrightarrow O + HCl$$

The weak hypochlorous acid is converted into very strong hydrochloric acid and the chlorinating bath becomes more acid.

In strongly acid solutions, nascent chlorine is liberated.

$$HOCl + HCl \rightleftharpoons 2Cl + H_2O$$

The nascent chlorine acts as an oxidizing agent, and the end-products and increase in acidity are the same.

Oxidation or chlorination of nitrogen and aromatic groups in the wool, or of other reagents in the bath, may further obscure the course of the reaction.

G. Controlling the Chlorination Reaction

The pH of the chlorinating bath is a most important variable, having a profound effect on the reaction with wool. On the alkaline side there is less likelihood of damage, but shrinkage-control is poorer and the wool becomes yellower.

Shrinkage control and whiteness are improved at lower pH, but the possibility of fiber damage increases. The reaction on the *acid* side is very rapid and it is difficult to control uniformly. Free chlorine is liberated to the atmosphere, making it impossible to estimate how much is used in the desired reaction, and difficult to obtain reproducible results.

Harris *(14)* has shown the effect of pH on oxidation potential, alkali solubility, weight loss, and area shrinkage. On the alkaline side best results appear to be obtained between pH 8 and 10.

Fig. 3 illustrates the manner in which the oxidation potential (Eh), of calcium hypochlorite solutions varies with pH and with concentration. It will be noted that pH has a profound effect, and that the potential rise obtained by a decrease of one pH unit is about equal to the potential rise obtained by a tenfold increase in concentration.

Fig. 3 helps to explain some of the difficulties which have been observed in the past history of chlorination of wool. A certain definite oxidation potential is required to rupture disulfide linkages. If the potential is below this minimum value, all the chlorine can be exhausted in side reactions, chloramine formation etc., without breaking a single disulfide bond. If the potential is too high other reactions may occur, such as rupture of the main polypeptide chains in addition to the de-

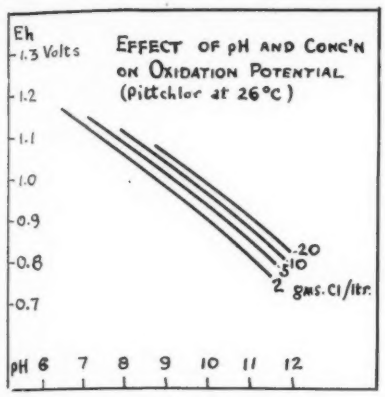


Figure 3—Oxidation Potential of Calcium Hypochlorate Solutions.

Voltage Developed by Gold vs. Calomel Electrode (Calomel Electrode Potential Added to Obtain Eh).

sired reaction. While it has been frequently stated that rapid chlorination is more effective because it limits the attack to the outside scale layer, it is just as likely that its effectiveness is really due to the increased oxidation potential at a lower pH.

Due to the effect of concentration on oxidation potential, it is evident that the gradual addition of hypochlorite will keep its concentration and hence its potential at a minimum, while addition of all the hypochlorite at once would give the maximum oxidation potential. Smaller liquor ratios would be more effective than larger ratios, since the higher concentrations would result in slightly higher potentials. These two effects may be used to increase or decrease oxidation potential as desired, depending on the pH of operation.

The rate of chlorination, and hence the uniformity of reaction, is successfully controlled at present by two methods:

(1) In alkaline, neutral or mildly acid baths by controlling pH, temperature, rate of addition and liquor ratio.

(2) In strongly acid solutions by addition of sulfonamides which form moderately stable chlorsulfonamides *(4)* or chlorsulfamates *(5)*.

H. Alkaline to Mildly Acid Chlorination Methods

The most favorable pH for the neutral and mildly acid chlorination processes lies in the range 4 to 10. Several investigators have employed buffers to adjust the bath to various pH values. Some prefer pH of 9 to 11 *(6)*, others pH of about 8. On the acid side pH has been buffered to 5.5 *((7)*, and in a British process the pH is adjusted at 4 to 5.5 *(8)*.

In some of these processes, by careful control of chlorinating conditions, damage to the wool fiber can be almost completely eliminated. However, disconcerting discrepancies occasionally arise in ability to control felting shrinkage.

In the most recent method of wet chlorination *(9)* wool is scoured and subjected to a special pretreatment, to make certain that its condition is uniform before chlorination begins. Thus results are uniform throughout the batch and from one batch to another *(10)*.

After the pretreatment, the wool is chlorinated with sodium or calcium hypochlorite in the cold (about 15° to 20° C.), using 3% to 8% chlorine based on the weight of wool. The exact amount of chlorine required depends on the type of wool and the yarn or fabric construction, and must be predetermined for each new type of material. Where a peroxide bleach is to be used, only about half as much chlorine is required.

This process is being used commercially on hosiery, yarn in skeins or packages, wool top and piece goods. As is the case with all chlorination processes, chlorine-resistant processing equipment is required. Stainless steel or wood dyeing equipment is generally satisfactory, but iron or brass fittings must not be allowed to come in contact with the wool.

Fig. 4 illustrates the effect of pH on rate of exhaustion of chlorine from calcium hypochlorite solution in contact with wool. In curve 1, the wool has been pretreated by the above method, so that pH drops during chlorination from 11 to about 7 or 8. The exhaust rate is slow enough to permit uniform reaction with the wool, but stabilization is far greater than would otherwise be achieved in this pH range.

In curve 3 the wool has been pretreated and chlorinated in a solution buffered at pH 8.5. Exhaustion is about twice as rapid, and shrinkage control is somewhat better than in sample 1; however, unless the machine employs excellent liquor circulation, there is possibility of uneven chlorination.

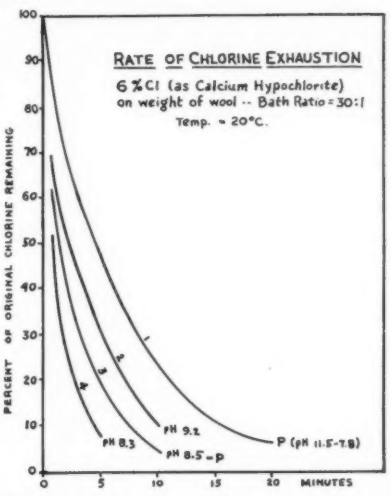


Figure 4—Exhaustion of Chlorine from Alkaline Calcium Hypochlorite Baths.

Samples 2 and 4 were treated in buffered baths at pH 9.2 and 8.5 respectively, without pretreatment. Under these conditions it is extremely difficult to obtain uniform results, and shrinkage control is not as good as in samples 1 and 3.

Table I illustrates the shrinkages of flannel samples treated as described above, after six consecutive half-hour washes with soap at 100° F. (CS 59-44). This was followed by two washes with a kerylbenzene sulfonate detergent in place of soap. It will be noted that shrinkage is much greater with the synthetic detergent. In the case of sample 1, good stabilization to synthetic detergent was obtained by increasing the amount of chlorine slightly. Sample 3 was quite satisfactory to both types of laundering. (Sample 4 was washed only with synthetic detergent).

I. Effect of Laundering with Synthetic Detergents

The effect of substituting synthetic detergents for soap in laundering is startling enough to warrant a brief digression at this point. Synthetic detergents have been advertised widely for use in home and commercial laundries, and in many

TABLE I
Warp Shrinkage of Chlorinated Wools

		Soap Washes						*Synthetic Detergent Washes*	
		1	2	3	4	5	6	1	2
P	11.5-7.8	0	0	0	0	0	—1.8	—6.6%	
	9.2	—1.2	—1.5	—1.5	—1.5	—1.8	—2.7	—7.4	
P	8.5	+1.0	0	+1.0	+1.8	+1.8	+1.2	0	
	8.3*	—	—	—	—	—	—	—2.5	—4.0

(* No soap washes run on this sample). (P indicates special pretreatment).

cases specifically for the *washing of wool.* Nevertheless we find that when wool is washed in a wash wheel with certain synthetic detergents in place of soap, felting takes place several times as fast, and some chlorinated woolen fabrics which would be quite satisfactory for a large number of soap washes, felt noticeably in a single wash with kerylbenzene sulfonate. Despite the fact known for a long time *(15)* that sulfated alcohols and amides are powerful fulling-agents, this effect still seems to cause some surprise.

The true value of the synthetic detergents for wool washing must obviously lie in the fact that wool can be washed at a lower temperature and in a shorter time, with less mechanical action than is required with soap. The danger however, is that with packaged synthetic detergents in common household use, the housewife may throw woolens into the washing machine and find they shrink badly, even though advertised as washable. Obviously garments which are stable only to soap washing should be labeled with instructions to that effect, and it might be well to avoid encouraging the housewife to wash wool in a washing machine. Nevertheless, since no one can really predict just what the housewife will do to shrink-resistant wool garments, the only safe procedure is to carry out laundering tests with synthetic detergents as well as soap.

J. Acid Chlorination Process

An outstanding solution to the problem of obtaining uniform acid chlorination with no yellowing is the conversion of hypochlorite to sodium chlorsulfamate before adding to the bath *(5, 10, 11):*

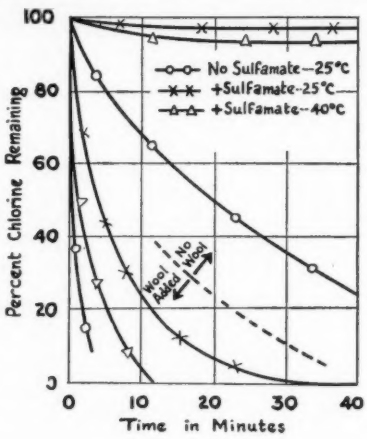
$$NaOCl + H_2NSO_3Na \longrightarrow Cl{-}NH{-}SO_3Na + NaOH$$

$$2NaOCl + H_2NSO_3Na \longrightarrow \begin{matrix} Cl \\ \diagdown \\ N{-}SO_3Na \\ \diagup \\ Cl \end{matrix} + 2NaOH$$

Either a mono-chlor or di-chlor derivative may be prepared. In actual practice, 1½ mols of chlorine are combined with 1 mol of sodium sulfamate.

Chlorsulfamate solutions are quite stable at pH of 1 to 2 in the absence of wool. In Fig. 5, the upper curves illustrate the great stability of chlorsulfamate baths at 25° C. and at 40° C., and this is contrasted with a loss of half of the chlorine in 20 minutes from an ordinary acidified hypochlorite bath.

When wool is added to the acidified hypochlorite bath, exhaustion of chlorine takes place in a few minutes, and uniform chlorination throughout the batch is most improbable. In the bath containing sulfamate, chlorine is exhausted more slowly over a period of about 30 minutes at 25° C., or 12 to 15 minutes at 40° C. Thus the temperature can be varied to control the reaction rate.



EFFECT OF SULFAMATE ON STABILITY AND CHLORINATION RATE AT PH 1-2

Figure 5—Exhaustion of Chlorine from Acid Baths Containing Sodium Sulfamate.

A small amount of formaldehyde is generally added to the bath in the acid sulfamate process to protect wool from degradation. Too much formaldehyde, however, affects the dyeing properties and hand of the wool, and reacts with the chlorine.

After exhaustion of the chlorine, the wool is antichlored with bisulfite, neutralized with sodium bicarbonate and dyed.

K. Alkali Solubility Determinations

Investigations of the alkali-solubility of wool before and after chlorinating were carried out during the war, and wearing qualities were in some cases found to vary inversely with alkali-solubility *(12).*

In general, it has been found that alkali-solubilities are much greater for acid processes than for alkaline processes. Certainly, where alkali-resistance is of importance, the alkaline process should be used. However, the relation between alkali-solubility and wearability may not be quite so clear. Even in cases of chlorinated wool of high alkali solubility, fabrics have shown virtually no tensile strength loss after a number of accelerated wash tests, and practical wearing tests on hosiery treated by the acid-chlorsulfamate process have shown no decrease in wear resistance.

The alkali solubilities of a 64s quality worsted top treated by the alkaline and acid processes described above are shown in Table II.

TABLE II

	Alkali Solubilities
Untreated Wool	14.0%
Alkaline Process	
4% Cl	17.8%
5% Cl	15.7%
Acid Process	
3% Cl	27.0%
5% Cl	30.2%

Summary

Various factors entering into shrinkage control of wool textiles have been discussed and an acid and an alkaline chlorination process, both in commercial use, have been described.

Acknowledgement

The writer acknowledges the invaluable work of Leo Beer, Mark Weisberg and Jonas Kamlet who have conceived and developed the processes described herein, and have assisted in the preparation of this paper.

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Discussion
(At Mid-West Section Meeting)

Chairman Jones: Mr. Shapiro will be glad to answer any questions you may have on this interesting subject.

Mr. Boxser: What chemical class of detergents causes the most rapid felting?

Mr. Shapiro: This subject has not yet been studied completely. In our tests, a kerylbenzene sulfonate, an amido-sulfonate, an amido-sulfate and a sulfated alcohol type all caused greater shrinkage than soap. Non-ionic detergents were not included in our study.

Mr. Hultberg: What was the pH range of the synthetic detergents used in wash testing?

Mr. Shapiro: The synthetic detergents were essentially neutral, pH 6 to 8. We suspected that the difference from the pH

of soap might be responsible, but addition of soda ash up to pH 10 to 11 had no effect.

Mr. Hultberg: Does the alkaline chlorination process yellow wool more than the acid process? What is the yellow color, and how can it be removed?

Mr. Shapiro: In general, the alkali processes cause more yellowing. The causes of yellowing have not been completely studied, and the formation of chloramines has sometimes been suggested. The discoloration does not vary directly with pH as implied in the technical literature, but depends on other factors. One factor is undoubtedly the grade of wool and thoroughness of preliminary scouring.

The yellow color is partly removed by the bisulfite antichlor. For true whites, further bleaching with peroxide or hydrosulfite is required.

Mr. Feit: Why hasn't the thioglycollate method been used for shrinkage control?

Mr. Shapiro: Harris' thioglycollic acid reduction of wool, followed by treatment with an alkylene dihalide to rebuild the broken linkages, has produced remarkable results. The price of thioglycollic acid is probably the principal reason why it has not been used.

Mr. Hultberg: I understand that the thioglycollic acid process imparts alkali resistance rather than shrinkage-control to the wool.

Mr. Shapiro: I believe Dr. Harris has reported both alkali-resistance and shrinkage-control for this process.

Mr. Meyer: What buffers are used in the alkaline process?

Mr. Shapiro: Any of the standard type buffers is satisfactory. Mixtures of polybasic acids and their salts are generally employed.

Mr. Jones: What about the dyeing properties of chlorinated wool?

Mr. Shapiro: When properly treated by the methods described, the dyeing properties are not greatly affected, and approximately the same shade is obtained on chlorinated as on unchlorinated wool. The New York paper in the October Sectional Contest indicated that the washfastness of really fast dyes is not affected, but that the less fast dyes suffer some reduction in fastness. Obviously, if wool is chlorinated to make it wash-resistant, then wash-resistant dyes should be employed.

Even though chlorinated and unchlorinated wool dye the same shade when handled separately, they should not be dyed in the same bath. Because of differences in affinity, two widely differing shades may result.

Chlorination may be carried out advantageously *after* dyeing if chlorine-resistant dyestuffs are selected.

Mr. Fleischmann: Which process gives the better hand?

Mr. Shapiro: The alkaline process generally results in a slightly softer hand than the acid process.

Mr. Feit: What is the Protonized Process?

Mr. Shapiro: The two processes which I have described in detail are known by that designation. The Protonized alkaline process is based on the special pretreatment, and results in much greater shrinkage control than would ordinarily be obtained at the same pH with no pretreatment.

The Protonized acid process is the chlor-sulfamate method. Both are the subject of patent applications.

Anonymous: Can these processes be applied in rawstock dyeing machines?

Mr. Shapiro: If liquor circulation is adequate, there is no reason why it cannot be applied on this type machine. It is already being applied on top-dyeing machines or on packaged-yarn dyeing machines.

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Outing Committee, New York Section

DR. HERMAN E. HAGER, Chairman of the New York Section, announces the following committee for the outing and golf tournament to be held on June 25th at the North Jersey Country Club, Pompton Lakes, New Jersey:

Patrick J. Kennedy—*Chairman.*
Hugo Todebush—*Golf.*
Robert L. Holoch—*Horseshoes.*
John H. Hennessey—*Finance.*
Claire W. Bendigo—*Press.*
Charles W. Dorn—*Dinner.*
Paul J. Luck—*Prizes and Awards.*

—♦ ♦—

Spring Meeting, Mid-West Section

THE spring meeting of the Mid-West Section was held at the Schroeder Hotel, Milwaukee, Wisconsin, May 1, 1948.

The afternoon meeting was called to order at 2:45 P.M., Elliott Morrill, vice chairman, presiding. Ninety-seven members and guests were present. Leonard Armstrong was introduced as the organizer of our sectional paper and complete cooperation with him was requested for the job ahead. The vice chairman then introduced our guest speaker, Dr. H. Luttringhaus of the General Dyestuff Corporation, who gave a talk on the "Influence of Chlorination on the Dyeing of Wool". He was assisted by James Flint. A great deal of interest in the presentation was evidenced by the number of questions asked and by those who stayed to inspect the exhibits. The meeting was adjourned at 3:40 P.M.

Dinner was served at 7:15 P.M. and 88 members and guests were present.

A short discussion followed concerning plans for the summer outing.

A nominating committee was appointed as follows:

Herman Boxser—*Chairman.*
L. B. MacFarland.
H. Thompson Latham.
Paul Soderdahl.
Louis Hoehn.

Leonard Armstrong, chairman of the technical committee, reported on work being done on the intersectional paper. He appointed the following committee members:

Bertil A. Ryberg, Leslie O. Meyer, Earl W. Scott, John E. A. Schroder, Merrill G. Morris, John C. Moertel, Paul Soderdahl, Clarence Wille, William F. McNamara.

The reading of the minutes and treasurer's report were dispensed with.

Our guest speaker of the evening was then introduced by the chairman. H. F. Clapham of the E. I. duPont de Nemours & Co. Inc., gave a very interesting talk on the subject of "The Dyeing of Spun Nylon, Filament Nylon and Nylon-Wool Mixtures".

Following questions and discussion from the floor, the meeting was adjourned at 9:30 P. M.

Respectfully submitted,
WILLIAM BOYD, *Secretary.*

Reports of Local Section meetings, papers and discussions should be sent to the National Secretary

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Advance notices of future meetings should be sent to the American Dyestuff Reporter

Lapel Buttons for AATCC Members

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These buttons bear the seal of the Association and may be purchased by members in good standing from the National Secretary at $2.00 each.

Crockmeters and Crockmeter Cloth

Revised Prices

Regular Crockmeter	$20.00
Regular Crockmeter (with automatic counter)	30.00
Duplex Crockmeter	50.00
Crockmeter Squares (500)	1.50
Crockmeter Cloth (96 x 100), by the yard	.75

Available from the National Secretary:

Dr. H. C. Chapin
Lowell Textile Institute
Lowell, Mass.

NEW TEST FABRICS

New test fabrics are available from Werner Klaas of Testfabrics, 224 West 35th Street, New York 1, N. Y.

Of the two sponsored by the Association, one is similar to the old Multifiber Fabric, but of firmer construction, containing filling stripes of Wool, Viscose, Spun Silk, Bleached Cotton, Celanese Acetate, and Nylon, assembled in 2 inch strips, with a waste stripe between repeats, in fabric 38 inches wide, at present price of $4.00 per yard.

The second contains Acetate, Wool, and Bleached Cotton, so woven as to show on its surface one inch squares of Acetate and Wool and two inch squares of Cotton. This is 54 inches wide, and is for the present priced at $3.50 per yard.

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CALENDAR

PHILADELPHIA SECTION
Meetings: September 24, November 12, January 28, 1949 (Kugler's, Philadelphia); Outing: June 11 (Atlantic City Country Club, Northfield, N. J.)

NEW YORK SECTION
Outing: June 25 (North Jersey Country Club).

PIEDMONT SECTION
Outing: June 18 and 19 (Ocean Forest Hotel, Myrtle Beach, S. C.).

NORTHERN NEW ENGLAND SECTION
Outing: June 11 (Merrimac Valley Country Club, Methuen, Mass.) Meetings: September 24 (Andover Country Club); November 19.

SOUTHEASTERN SECTION
Meetings: July 24 (Atlanta, Ga.); September 25 (Columbus, Ga.); December 4 (Atlanta, Ga.).

SUB-COMMITTEES
Meeting: June 7 (Hotel New Yorker, New York).

COUNCIL
Meeting: June 8 (Hotel New Yorker, New York).

RESEARCH COMMITTEE
Meeting: June 8 (Hotel New Yorker, New York).

NATIONAL CONVENTION
October 21-23 (Sheraton Bon Air Hotel, Augusta, Ga.).

SOUTH CENTRAL SECTION
Outing: June 4 and 5 (Whittle Springs Hotel, Knoxville, Tenn.).

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EMPLOYMENT REGISTER

This column is open for four insertions per year, per member, without charge. Blanks can be obtained from, and filed with, the Secretary of the Association, Lowell Textile Institute, Lowell, Mass. It is understood that these will be open to inspection by prospective employers who can obtain further information from the Secretary.

48-5

Education: B.T.C., Lowell Textile Institute.

Experience: Textile chemist and dyer, wool and cotton.

Age 40; married; references; will work anywhere in U. S.

48-6

Education: B.A. University of Pennsylvania, with graduate study.

Experience: Fifteen years, wool scouring, carbonizing, bleaching; synthesis of wetting agents, detergents, textile oils and sizes; shrink proofing, waterproofing, mildew proofing.

Age 36; married.

48-7

Education: Technical school, with additional college courses.

Experience: Textile chemist, with practical experience in dyeing and finishing of cotton and rayon, including resin finishes. Textile Technologist, U. S. Quartermaster Corps, during war. Good knowledge of Spanish and Portugese.

Age 26; married; references; will work anywhere.

• PATENT DIGEST •

Glass Fibers—Coloring by Coating Processes C, 4, 07

U. S. Pat. 2,428,302
(Owens-Corning-Fiberglas Co., Trowbridge, Sept. 30, 1947)

The first attempts to produce colored glass fibers were based on distributing pigments within the mass. Obviously many difficulties were met in these operations. The pigments alter the nature of the glass substance in a considerable amount. Therefore the range of permissible pigments is very limited. On the other hand a dyeing process according to the standard dyeing procedures gives only very superficial effects, bleeding off and not at all resistant to crocking. According to the present invention the glass fibers are coated with a dyestuff, dispersed with a film forming substance in aqueous solution and solubilized in a subsequent step. The film forming material is preferably a protein, for instance a plasticized glue, containing Sorbitol, corn syrup or the like. A suitable composition is said to be available in the trade under the name "Gresistoflex". It is advisable to apply the substance to the fibers during the filament forming process. One formula, especially recommended, reads:

2% of a dyestuff, setting with formaldehyde.
3% of Gresistoflex, setting with formaldehyde.
0.5% of a cation active agent (example: octadecyl- monoamine acetate)
2% formaldehyde
Balance: water.

It is also proposed to omit the formaldehyde in the formula and to spray in a second operation with formaldehyde solution. By insolubilizing the dyestuffs (types: "Amanil", "Calcoform", "Formanil", etc.) very equal coatings, better than any pigment dyed glass fibers are obtained.

References cited by the Patent Office are, amongst others:

U. S. Pat. 2,361,277 (Ciba): Stable emulsions are formed containing casein in the oily phase, aldehyde in the aqueous phase, for pigment printing.

U. S. Pat. 2,215,061 (DuPont): Coloring glass fibers is carried out by applying a pigment dispersed in a nitrocellulose-acetone-alcohol solution.

U. S. Pat. 2,193,818 (Japanese Company Kaisha): Sizing with a matured protein solution (soy bean protein), containing sugar and tartaric acid as a stabilizing agent.

Other References are:

(Reference is made to abstract to U. S. Pat. 2,394,493 in AMERICAN DYESTUFF REPORTER 1946, p. 336, assigned to the Owens-Corning-Fiberglass Co.)

U. S. Pat. 2,323,684 (Owens Corning): Teaches to prepare a coating composition for glass fibers by using a wax and a cation active substance as an emulsifying agent. The wax is merely used for lubricating purposes.

U. S. Pat. 2,339,928 (same company): Suggests incorporating metal oxides in the mass and to reduce subsequently these oxides to get the color effect of the dispersed metal.

U. S. Pat. 2,403,872 (same company): Applying a resinous pre-condensate and forming a thin film, covering the fiber in a subsequent curing process.

PAUL WENGRAF

Shrinkproofing Wool—Permanganate Treatment, Combined with Chlorine A, 2, 04

U. S. Pat. 2,429,982
(Stevenson Dyers Ltd., Stevenson-Raynes, Oct. 14, 1947)

The present patent states that shrinkproofing wool cannot be carried out in too highly acid baths because of the strong substantivity of wool to the acid halogen solutions. No uniform result can be expected in working this way. On the other hand hypochlorite solutions act much slower in a bath, adjusted to a pH 7-8 but no chlorinating action occurs. The halogen acts merely as an oxidizing agent and no appreciable non-felting effect can be attained. It has been found that by combining halogen—containing nitrogen compounds or chlorine with permanganate, the anti-shrinking effect is substantially improved, and wool does not lose any of its attractive properties such as touch, isolation, etc. Nitrogen-chloro compounds are amongst others: nitrogen trichloride, monochloroamine, etc. In contradistinction to the hypochlorites they should be applied in a separate bath, i.e. not in admixture with permanganate. An example is: First bath: Treating wool at 40° C. in a 0.1% potassium permanganate solution.

Second Bath: Rinsing and treating the material in a dilute solution of sodium hypochlorite.

Third Bath: The manganese discoloration is destroyed by bisulfite.

References:

The patent may be identical with the older Brit. P. 569,730. U. S. Pat. 2,311,507 (1943) (Du Pont): the chlorination of wool is carried out with organic, non-aqueous solutions of a compound evolving chlorine in contact with moisture (Example n-chloro-p-toluene sulfonamide = Chloramine T). Combining of chlorine and persalts has often been proposed for improving bleaching operations but not for shrinkproofing wool (to compare U. S. Pat. 2,048,991, Boehme, Ger. Patent 615,680, I.G.-Waibel and U. S. Pat. 1,908,481 Kauffmann).

Reference is made to Brit. Pat. 586,020 (Wolsey Ltd.) abstracted in AMERICAN DYESTUFF REPORTER, 1947, p. 615

Scour Water from Wool Processing—Clarifying H, 6

U. S. Pat. 2,429,868
(Fields Point Mfg. Co., Campanella, Oct. 28, 1947)

For wool processing plants a serious problem consists of rendering wool scour waters sufficiently clear and odor free to be discharged in the sewer system. Raw wool which passes through a series of scouring bowls contains dirt, grease, albuminous or generally protein matter, manure, burrs and so on. According to the present invention the overflow of the scouring bowls is conducted to a reaction chamber and treated there with a calcium hypochlorite solution. During the reaction heavier and lighter solids separae out. The light compounds are skimmed off from the discharge end of the reactor, passed to a mixing tank, heated at 180-190° F. and from there to a system of centrifuges. After two times centrifuging a high quality grease is obtained. The heavier solids and the liquid overflow to a settling tank. The grease is recovered after settling by an acid treatment and by filtering through a press. Calcium hypochlorite is fed from a supply tank. The pH in this operation has been measured at 7.8.

References cited by the Patent Office are amongs others:

U. S. Pat. 2,070,286 (Louisville Drying Machinery Company): Distillery slop is treated at its iso-electric point with a hypochlorous precipitating agent to recover the suspended grain elements.

U. S. Pat. 1,418,013 (Scaife & Sons—Newman): Clarifying water by treating it with an alkaline bleaching agent (hypochlorite of lime) thereupon with a coagulant (aluminum sulfate) and finally with thiosulfate to remove excess chlorine.

Other reference:

Textile Colorist 1942, pages 124 and 132 states that the chemicals mostly used in clarifying waste waters coming from wool mills are sodium sulfate, sodium aluminate or both.

Crinkled Fabrics—Resins Printed as Resists D, 1

U. S. Pat. 2,429,935
(Cranston Print Works, Jones-Stackpole, Oct. 28, 1947)

A Plissé surface is usually given to a fabric by printing thereon spaced bands of gum or another resist and passing the cloth through a bath of a 20% caustic in the cold. The gum resists protect temporarily the printed portions from shrinkage and causes thereby the formation of crinkles by the shrinkage of the neighboring bands. This process requires, as the specification explains, an abnormal shrinkage for instance by means of strong alkalies. The present invention has found a shrinkage resist and a shrinking method whereby the material loss of yardage (occurring in former processes) can be avoided. The problem has been solved by printing the cloth, a non-crepe cloth or a fabric of normally twisted yarns, in a *stretched condition* with a hardenable resin, by hardening or insolubilizing this resin resist and subjecting the cloth to a normal slack laundering or Sanforizing operation which shrinks the non printed areas and causes the resist prints to crinkle. The easiest way to bring about this stretched condition is to lengthen the cloth in a standard bleaching operation in the rope. The resist may consist of a polymerizable resin of thermoplastic or thermosetting character which is set or insolubilized in the known way. For example a cloth, 3000 yards long, gains 20% in length after bleaching and it is narrowed in width at the same time from 38 to 32 in. It is printed *in this condition* (as usual in other printing operations too) with a melamine formaldehyde precondensate, thickened with starch or sodium alginate, to which ammonium chloride is added as a catalyzer. In drying, the melamine aldehyde compound polymerizes and hardens. It is indicated that the quantity of the resin fixed in any curing operation is too small to make the printed areas permanently stiff but it is sufficient to hold the fibers covered by the printing paste firmly bound together even in a subsequent glazing process on a heated calender. The next step comprises shrinking in a laundering operation and drying without subjecting the cloth to any tension in longitudinal direction. In this way "abnormal shrinkage operations" i.e., strongly alkaline or acid treatments are avoided.

References cited by the Patent Office are amongst others:

U. S. Pat. 2,319,903 (Sayles Finishing): Proposes to produce a relief print by covering a part of the cloth with alkylcellulose, for instance with "Colloresin", and shrinking with caustic.

U. S. Pat. 2,349,914 (Heberlein): A resist under (parchmentizing) cold sulfuric acid consists of varnish-fixed cotton flock.

U. S. Pat. 2,200,389 (Celanese): Printing a resist of acetone soluble acetate of cellulose on normal crepe fabrics, consisting of highly twisted yarns and aftertreating the fabrics as usual with hot lye.

U. S. Pat. 2,022,391 (Celanese): Crinkled crepe-like fabrics are prepared by interweaving yarns of cellulose acetate with *stretched yarns* of regenerated cellulose. The woven fabrics are scoured, dyed and dried. The pre-stretched yarns shrink and cause the non-stretched cellulose acetate yarns to crinkle.

Other references related to this subject are:

Crimping of fabrics by pre-printing an Arabic gum or British gum resist and padding in caustic lye of 63° Tw. for 3-4 min. has been known for a long time according to "Principles and Practice of Textile Printing" by Knecht and Fothergill (1916). This style is said to be due to Mercer (p. 850). Later on the resists on crimping have been combined with various other printing pastes and dyed in different ways. Combined parchmentized (sulfuric acid) and Plissé-effects are described in Heberlein's U. S. Pat. 2,382,416 (1945). Brit. P. 481,459 (same company) recommends pre-printing of resins as resists under sulfuric acid treatments.

Bleaching Polyacrylonitriles B, 5

U. S. Pat. 2,432,447
(Du Pont, Scheiderbauer, Dec. 9, 1947)
U. S. Pat. 2,432,448
(Du Pont, Richards, Dec. 9, 1947)

Polymers of acrylonitriles have acquired an increased technical importance as a material for manufacturing yarns. The first of these patents protects a bleaching process of polyacryle threads (dry or wet spun) by treating the material in an aqueous hypochlorite solution containing 0.05-1% available chlorine. According to an example, the polymer substance is dissolved in dimethylformamide and extruded through a spinneret into a cell, heated at 130° C. The collected and dried yarn is wound upon a bobbin and bleached in a hypochlorite solution of a chlorine content as indicated here above and adjusted to a pH of 2.1. The bleaching operation is carried out under pressure in an autoclave at a temperature between 90-150° C. (thus in a steam atmosphere). The time for treating this material in the bleaching solution is limited with 30 minutes. The second named patent suggests treating the yarns in a similar way with hypochlorous acid or hydrogen peroxide as long as the yarns are *still in the gel state* (in contradistinction to the content of the first mentioned patent where *dried* yarns are subjected to the procedure). The gel state is defined as the condition prevailing before the gel structure has collapsed, i.e. while the yarn still possesses a large amount of coagulating bath liquid (about 100% calculated from the acrylonitrile substance).

References citel by the Patent Office are amongst others:

U. S. Pat. 2,295,660 (Hubert et al.): The heat stability of polyvinyl yarns is improved by heating them for about 2 hours in moist chlorine. The process is not intended for bleaching purposes.

U. S. Pat. 2,292,737 (Bloemer et al.): Polymers of the synthetic rubber type are chlorinated at 60-125° C. for obtaining higher chlorinated compounds.

U. S. Pat. 2,021,763 (Rohm & Haas): Describing the chlorination of acrylic acid polymers, dissolved in organic solvents by a stream of gaseous chlorine.

U. S. Pat. 2,116,210 (Du Pont): Treating regenerated cellulose yarns with solutions of about the same concentration and pH-value as described in the present patents.

Creaseproofing Textiles—Wet, Free Shrinking Treatment G, 2, 04

U. S. Pat. 2,433,620
(United Merchant & Manufacturers, Monsaert Dec. 30, 1947)

Generally, creaseproofing finishes comprise an impregnation with artificial resin precondensates, subsequently insolubilized by a curing treatment. The present process differs from known methods therein that the fabrics are subjected to a *tumbling and beating action* in a drum, internally provided with bars or baffles. During the entire operation consisting of *(1)* desizing, *(2)* scouring, and *(3)* rinsing, this tumbling and beating action has to be continued. The fabrics rotate freely in a foraminous cylinder without using any tensioning device. This rotating cylinder is placed within another fixed cylinder, containing the treating liquids. The baths are maintained at elevated temperatures but still below the boiling point. The result of the process is characterized by the fact that an increase in weight of 12.5% was obtained while normally treated and finished fabrics produced only an increase of about 0.3% calculated on the same area). It has to be emphasized particularly that a fabric when folded and

crushed under a weight does not form sharp creases but rather soft and full folds. It has quite another appearance than the original material. This fact could only be explained by a more dense texture and wool-like surface and also possibly by the fact that the "apertures of the cloth are filled with interlacted fibers."

References cited by the Patent Office are amongst others:

U. S. Pat. 2,325,544 (Redman): Protecting a process and an apparatus for detensioning fabrics and restoring the mechanical conditions of the fibers by allowing an extensive shrinkage. The fabric is rumpled lengthwise and widthwise in a tension-free state in the presence of moisture and heat.

U. S. Pat. 2,191,977 (Kalle & Co.). Describing an enzyme treatment for spun rayon (staple fibers).

U. S. Pat. 1,717,992 (Meliana Corp.): The strength of cotton fibers is said to be increased by a highly concentrated lye at temperatures between 60-100° C.

U. S. Pat. 1,679,767 (Gminder): Cotton fabrics acquires a wool like appearance by a raising operation followed by a treatment with strong alkalies with or without tension.

Earlier work done in this field:

The principle of the present invention seems to consist of a shrinking process, carried out by a treatment in a non-tensioned condition. Generally this type of shrinkproofing or creaseproofing finishes was based on alkaline pretreatments ("tensionless mercerizing") as described, for instance, in Drechsels Brit. P. 437,485 and in many other publications. The innovation contained in the present patent comprises a simple detergent treatment at moderate temperatures and under similar mechanical conditions. The absence of harsh chemical agents may explain the effect of a full and soft hand. Brit. P. 375,945 (Chadwick) claims to obtain by mechanical means a creaseproofing effect by treating the fabric in completely relaxed condition in a steam atmosphere whereby the fabric can freely shrink in length and width.

TRADE NOTES • NEW PRODUCTS

• Schollerized Wool Shrinkage Control

Scholler Bros., Inc., Collins & Westmoreland Sts., Philadelphia 34, Pa., introduced the Schollerized process of wool shrinkage control at a demonstration in their laboratories on May 12th. The demonstration was under the direction of Frederic C. Scholler, President, Lloyd O. Koons, Vice-President and Treasurer, and Dr. Edwin C. Dreby, Research Chemist.

After a thirty minute washing with soap in a laundry machine, the Schollerized wool apparently shrank but a negligible amount whereas untreated woolens, washed at the same time, shrank and matted so much that they were no longer serviceable.

The wool is treated by a controlled chlorination process, and it is claimed, is practically shrink-proof for the life of the fabric. Matting is said to be completely eliminated so that the wool garment maintains its original size with all of its natural qualities of resilience, elasticity, softness, texture and comfort. It is stated that no foreign material is added to the wool and nothing is taken from it. It was pointed out during the demonstration that the process permanently dekinks the fibers and thus eliminates subsequent matting or felting during washing.

Garments that are knit or woven of Schollerized wool require no particular care in washing, it was stated. Assuming the colors are fast, the Schollerized wool garment may be washed with the exercise of no greater care than any other colored or dyed washable wearing apparel. Blocking to shape is advisable after washing.

"Schollerized wool is unaffected by rubbing or tubbing", a company statement says. "Not only is it unaffected by rough tub or washing machine action but the same properties that control the shrinkage also make it difficult for dirt to successfully cling to wool. The elimination of felted dirt-traps in the wool fiber strands permits ordinary soap and water to do a better-than-ever job. Fast colors stay brighter and the whole garment retains its new-like appearance for the life of the fabric.

Dr. Edwin C. Dreby, Research Chemist for Scholler Bros., Inc. comparing untreated wool fabric with Schollerized wool fabric after 30 minutes washing test.

"No company, considerate of its own integrity and of its responsibility towards its customers and the ultimate consumer, may honestly say that any shrinkage control process renders any fabric 'shrink-proof' in the ultimate technical sense. Shrinkage occurs in every fabric, even though such shrinkage may be infinitely small—negligible from a practical viewpoint.

"We sincerely believe that the Schollerized process of wool shrinkage control renders wool fabric as nearly shrink-proof as it is possible to attain. After scores of washings, the original Schollerized knitted or woven size was retained almost exactly, in some cases showing a shrinkage factor of less than 1 percent. This applies to any product made of wool—socks, sweaters, women's sport clothes, men's shirts and jackets, blankets, baby wear and the host of other wool products that will now take a new lease on life because 'made to size, it will stay to size' for the life of the fabric."

The Schollerize process is adaptable to ordinary mill equipment and may be applied to raw stock, yarn or piece goods. It may also be used on mixtures of wool and other fibers. The process is available to any interested mill and may be sold under the mill label or under the Schollerize label. When the Schollerize label is used the company will insist upon a quality control plan to be conducted under its supervision and license agreement.

• Annual Meeting, CATCC, Quebec Section

The annual meeting of the Quebec Section of the Canadian Association of Textile Colorists and Chemists was held on Saturday, April 17th, at the Mount Royal Hotel, Montreal. Over 300 mem-

bers and guests, including textile men from the United States and Britain, attended.

President H. K. Torpey, Dominion Textile Co., Ltd., presided at the business meeting in the afternoon and said that the membership had increased to 185, there had been more technical meetings, a textile library had been established at Sir George Williams College, and classes in the technology of fibers and dyeing had again been sponsored at this institution. Student visits to the Belding Corticelli and The Montreal Cottons, Ltd. mills had been arranged and further visits would be sponsored. Officers were elected as follows for the 1948-49 season:

Honorary President—E. Bradley.
Past President—H. K. Torpey.
President—F. D. Wallace.
1st Vice-President—G. Wills.
2nd Vice-President—S. W. Milnes.
Council—W. W. Metcalf.
W. H. Pover.
E. Sewell.
L. J. Sheps.
J. Shower.
W. Stanfield.

Delegates to National Council—F. J. Knight, G. C. Taylor, F. D. Wallace, H. K. Torpey (Alternate).

Delegates to Federation—H. K. Torpey, W. King, F. J. Knight, F. D. Wallace (Alternate).

Secretary-Treasurer—R. W. Redston.
Auditor—Hugh B. Savage.

The annual dinner was held in the evening. Officers and guests at the head table with President Torpey included:

E. P. Carney—*President, Textile Technical Federation of Canada.*

F. D. Wallace—*President-Elect, Quebec Section, CATCC.*

G. Bruck—*Vice-President, Bruck Mills, Ltd.*

Myron D. Reeser—*American Dyestuff Reporter.*

R. W. Graham—*President, Ontario Section, CATCC.*

Ernest Bradley — *Honorary President, Quebec Section, CATCC.*

C. P. Atkinson—*Spooner Engineering Co., England.*

William King—*Canadian Textile Journal.*

W. G. Hicks—*General Manager, Belding Corticelli, Ltd.*

Dr. J. M. McIntyre—*Resident Surgeon, Queen Mary Veterans Hospital.*

R. W. Redston — *Secretary-Treasurer, Quebec Section, CATCC.*

W. W. Spooner—*Spooner Engineering Co., England.*

Dr. K. E. Norrie—*Principal, Sir George Williams College.*

A. C. Salter—*President, Textile Sales Ltd.*

F. D. Wallace
President-Elect, Quebec Section CATCC

Don Lee—*Nylon Division, Canadian Industries, Ltd.*

E. F. King—*President, Dominion Burlington Mills, Ltd.*

J. G. Sutherland—*Manager, Canadian Cotton Ltd., Cornwall.*

S. F. Toupin—*Director, Ecole Technical de Saint Hyacinthe.*

A. T. Oneson—*Recreational Supervisor, Queen Mary Veterans Hospital.*

R. F. Brown—*Secretary-Treasurer, Ontario Section, CATCC.*

No formal speches were delivered at the dinner. An appeal for funds for the Queen Mary Veterans Hospital resulted in a collection of $300. During the evening a variety show was presented by the entertainment committee under the chairmanship of James A. Clough.

On the preceding evening two prominent British textile men, W. W. Spooner and C. P. Atkinson of the Spooner Engineering Company of Yorkshire, England, spoke on the relationship of scientifically designed preparation, dyeing, drying, and finishing machinery to the efficient handling of textile fabrics in general and the shrinkage control and dimensional stability on spun rayon in particular.

Federation Officers

At a business meeting on Saturday, the following officers were elected by the Textile Technical Federation of Canada:

President—Edward Carney.
1st Vice-President—W. J. Buck.
2nd Vice-President—H. K. Torpey.
Honorary Secretary-Treasurer—P. W. Webb.
Assistant Secretary-Treasurer—V. Grauthan.

• 5th Ave. Sales Offices, Dye Plants

Consolidated sales and executive offices for three dye plants of New York and Philadelphia have been opened at 230 Fifth Avenue, it is announced by Jesse M. Schneider, Secretary-Treasurer of all three plants: A. M. Townson & Co., Inc., of Philadelphia; General Rayon and Silk Dyeing Corporation, and Decorative Specialty Dyeing Corporation, both of New York.

Norman Weil has been appointed New York representative for the A. M. Townson Co. and will make his headquatrers at the new office. Clive Steinman, formerly with Cohn-Hall-Marx Co. has been named to represent General Rayon and Silk Dyeing Corporation in the New York area.

Expanded facilities are also announced by all three firms. A new smallwares department with special machinery for handkerchiefs and decorative linen has been set up by General Rayon and Silk Dyeing Corporation under the direction of Albert Muller. New carbonizing equipment for woolens and gabardines has been installed by A. M. Townson Company. Additional warehouse space has been acquired by Decorative Specialty Corporation.

• Unisize

Announcement of a completely new type acetate and viscose warp size has just been made by Nopco Chemical Company, Harrison, N. J. Known under the trade name "Unisize" this new polymer is said to represent a most advanced development in warp sizing. It is a single, one-product size which acts as binder, softener, penetrant, lubricant, humectant and defoamer. It is stated that one of its most outstanding advantages is that it completely eliminates the need for auxiliary additives to the size.

Physically, it is a free-flowing, non-sticky, granulation which, in appearance, is very much like light brown sugar. It is stated that it is extremely soluble and dissolves readily in hot water with simple stirring. No lumps and gelling are said to occur to prevent even distribution of the size on the warp.

It is said to create a strong bond, form a flexible and non-tacky film, provide maximum filament protection, and eliminate static during slashing and weaving. A minimum of shedding occurs in the loom.

• Amalgamated Representative

Amalgamated Chemical Corporation, Philadelphia, has appointed Walter L. McNab sales representative for Tennessee and the northern part of Alabama and Georgia. The appointment became effective March 1, 1948. Mr. McNab has been associated with the Textile Industry in the sale of chemicals and dyestuffs for a number of years. He will make his headquarters at 2657 Magnolia Avenue, Knoxville 15, Tennessee.

• Resigns from IRI

Raymond B. Seymour, Chairman of the South Central Section of the AATCC, has resigned his position as director of the Industrial Research Institute of the University of Chattanooga. Before joining this organization, three years ago, Dr. Seymour was a research group leader for the Monsanto Chemical Co.

• ACCL Annual Meeting

The annual meeting of the American Council of Commercial Laboratories will be held June 18-20, at Colorado Springs, Colorado. The convention will have special significance this year because of the part the members of this Council will be called upon to play in promoting the standards and quality of merchandise furnished Europe under the Recovery Program. The Colorado Springs meeting will devote the major part of its discussion to the intricate problems involved in testing and outlining technical phases of international contracts.

• Joins Golwynne Chemicals

John V. Reilly has joined the Golwynne Chemicals Corporation. Golwynne handles a general line of industrial chemicals, dyestuffs, dye intermediates and pharmaceuticals. Mr. Reilly was formerly sales manager for the Bendix Chemical Corporation.

• Apex Chemical Addition

The Apex Chemical Co., Inc., 225 W. 34th St., New York City, through its President, Herman Rothstein, announces that a contract for about $80,000.00 has been awarded for a new addition to its manufacturing buildings at its plant in Elizabethport, N. J.

This is in line with the company's program of expanding its facilities for the manufacture of industrial chemical products for the textile, leather, paper and allied industries.

• Grasselli Appointments

The Grasselli Chemicals Department of the Du Pont Company announces the appointment of Howard J. Smith as assistant manager of its resale chemicals section in Wilmington and of Henry F. Koll as manager of its district office at Charlotte, N. C.

Mr. Smith was associated with the Grasselli Department for a period of years as branch manager and assistant district manager at Philadelphia before going to Charlotte as district manager in 1937. He comes to Wilmington from that post.

Mr. Koll joined the Grasselli Department at its Cleveland laboratory in 1932 and worked in various technical and manufacturing operations at the Cleveland plant until 1943 when he transferred to the plant at Grasselli, N. J. In 1944 he became a technical service representative, with headquarters in Wilmington, and from 1945 to 1948 he worked out of the Philadelphia sales office.

• U. S. Testing Appointment

The United States Testing Company, Hoboken, N. J., has announced the appointment to its Research Staff of Benjamin S. Van Zile, above, well-known authority on soaps and detergents. Mr. Van Zile, for many years, was associated with the Colgate Palmolive Peet Co., as Supervisor of the Analytical Research Division of the Development Department, specializing in synthetic detergents, soaps and cosmetics. He also conducted studies on the detergent properties of rosin for the Hercules Powder Company, as well as evaluations for water solubility emulsifying properties.

He is a member of the Committee on foreign correspondence of the American Federation of Scientists, currently engaged in the dissemination of information on atomic control to those scientists who are cut off from accurate information by some of the foreign governments.

• Westvaco Chemical Corp.

In recognition of the expanded scope of the Company's activities, stockholders of Westvaco Chlorine Products Corporation, New York, voted at the Annual Meeting on April 28, 1948 to change the corporate name to Westvaco Chemical Corporation.

While "chlorine products" are still an important part of its output, acquisitions and plant expansions have made Westvaco one of America's largest producers of barium and magnesium chemicals; bromine and brominated compounds; organic insecticides, fumigants and agricultural chemicals; phosphates and other chemical specialties.

• AIC Officers

The American Institute of Chemists elected the following officers at its Silver Anniversary Meeting, May 7, 1948 at the Waldorf-Astoria, New York:

President, Lawrence H. Flett, Director, New Products Division, National Aniline Division, Allied Chemical and Dye Corporation, New York, N. Y.

Vice President, Dr. Raymond E. Kirk, Head, Department of Chemistry, Dean, Graduate School, Polytechnic Institute of Brooklyn, Brooklyn, New York.

Secretary (re-elected), Dr. Lloyd Van Doren, Chemical Consultant, patent cases, Watson, Bristol, Johnson & Leavenworth, New York, N. Y.

Treasurer (re-elected), Dr. Frederick A. Hessel, President, Montclair Research Laboratories, Montclair, New Jersey.

Councilors, Dr. M. L. Crossley, Director of Research, American Cyanamid Company, New York.

Dr. James R. Withrow, Chairman, Professor Chemical Engineering, Ohio State University, Columbus, Ohio.

John M. McIlvain, Administrative Supervisor, Research and Development Department, The Atlantic Refining Company, Philadelphia, Pennsylvania.

• UPDW Exhibit

Miniature working models of dyeing and finishing equipment will be featured in the United Piece Dye Works exhibit at the Textile Exposition at Grand Central Palace from June 7-12.

Equivalent to a trip through a full-size finishing plant, the models will demonstrate the following processing steps: (1) boil-off, (2) dyeing, (3) moisture extraction (4) printing, (5) application of special finishes, such as Unidure, for permanent crease-resistance and Durasec, for durable water-repellency, (6) drying, (7) tentering.

In conjunction with the model machinery, fabrics in progressive stages of finishing and printing will be displayed.

The United family of special finishes—Unidure, Unifast, Durasec, and Airfast—will be represented by garments, especially made for the exhibit, which will visualize the particular merits of each finish.

Illuminated Kodachromes of current styles in Unidure fabrics and a background display of printed and plain United-dyed fabrics will also be included.

A photographically illustrated booklet entitled "United Finishes at Work," swatched with samples of fabrics in United's special finishes, will be distributed to visitors.

• General Aniline Staff Additions

Recently added to the staff of the Central Sales Development Department of General Aniline & Film Corporation were the following:

D. A. Beadell and Dr. D. E. Sargeant, transferred from the Central Research Laboratory;

William M. Shine, transferred from the Development Department;

Dr. Aubrey H. Nease, formerly with Phillips Petroleum Corporation;

C. F. McKenna, Jr., transferred from the Development Department;

Henry C. Speel, formerly with Atlas Powder Company.

The expanded department is under the general supervision of Cary R. Wagner, General Aniline Vice President in Charge of Research and Development, and Dr. Philip M. Kirk has been named Department Manager. Dr. Kirk has been active in General Aniline & Film Development Department work since 1943.

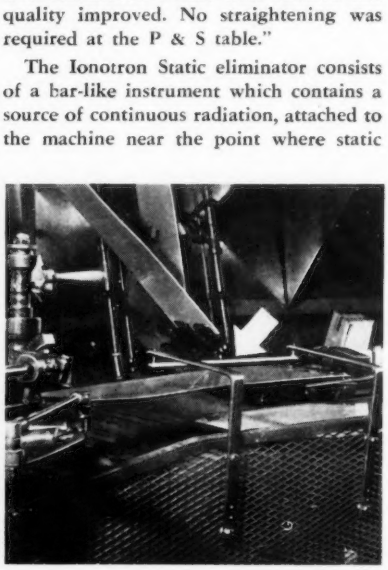
• Ionotron Static Eliminator

A new unit, the Ionotron* static eliminator, is said to have been successfully applied to Proctor & Schwartz automatic boarding machines to eliminate the problem of static electricity in boarding sheer nylon hosiery. It is stated that it operates entirely without electrical connections or moving parts, and for all practical purposes its service life is permanent.

The Ionotron, a product of the U. S. Radium Corporation, 535 Pearl Street, New York City 7, has been applied to end static problems on wool cards, coating machines, printing presses, packaging machinery and the like. An application has now been made for the Pohatcong Mills, Washington, N. J., on six automatic boarding machines. Particularly in the production of 15-denier nylon hosiery, static effects on the P & S boarding machines in this plant were said to be snarling production.

Ionotrons were installed on six of the automatic boarders and results were said to be highly successful. As a Pohatcong official said: "All difficulties due to static were eliminated. Mends were reduced and quality improved. No straightening was required at the P & S table."

The Ionotron Static eliminator consists of a bar-like instrument which contains a source of continuous radiation, attached to the machine near the point where static charges are generated. The radiation ionizes the air, making it a good conductor of static electricity. Static charges flow off through the ionized air to the eliminator, and from there harmlessly to ground. Another feature of the Ionotron is said to be its long life. It is claimed that the action of the Ionotron is permanent, as the active source loses only 50 per cent of its generating power in 1,500 years.



Ionotron Static Eliminator Installed on a Boarding Machine in the Pohatcong Mills, Washington, N. J.

The Ionotron has been placed at a point where the hose leaves the boards.

* Reg. Trade Mark.

• Joins Ultra Chemical

Fred H. Buck, former president of Seaboard Distributors, Inc., who has become Industrial Sales Manager for Ultra Chemical Works, Inc., Paterson, New Jersey. Mr. Buck has been actively associated with the chemical business throughout all of his business life, having begun in 1935, when he joined Merck & Company immediately after leaving Yale University. Mr. Buck is familiar with the products of the Ultra Chemical Works, for the company with which he was formerly associated had been acting as Ultra's selling agent, distributing chemical specialties throughout the United States and foreign countries.

• AIC Medal

Scientists today are taking a new role in the industrial leadership of America, Dr. Charles Allen Thomas of St. Louis said in New York on May 7th upon receiving the Gold Medal of The American Institute of Chemists.

Speaking at the Institute's Silver Anniversary Dinner, held jointly with the New York Section of the American Chemical Society at the Waldorf-Astoria, Dr. Thomas said that scientists forced into administrative positions because of the complexities of modern industry must acquaint themselves not only with the unchanging laws of chemical reactions but also with the everchanging aspects of human behavior. Dr. Thomas is President of the American Chemical Society and Executive Vice-President of Monsanto Chemical Company. He recently was honored by being elected to the membership of the American Academy of Sciences.

Dr. Foster D. Snell, retiring president of the Institute, presented the medal to Dr. Thomas citing him for his "outstanding work in the development of atomic energy, leadership in research, administrative ability and encouragement of basic research."

A short business meeting of the New York Section of the American Chemical Society was conducted prior to the medal presentation by the Section's chairman, Dr. H. T. Clarke, professor of biochemistry, College of Physicians and Surgeons, Columbia University.

Francis J. Curtis of St. Louis, Monsanto vice-president who has been closely associated with Dr. Thomas, spoke on the career of the medalist.

• New UPDW Printing Dept.

The United Piece Dye Works announces the opening of a roller printing department at its plant in Los Angeles, California. According to the United Piece Dye Works, this is the first completely equipped roller printing plant on the West Coast. Rayon crepes, Bemberg sheers, rayon jerseys, spun rayons, as well as novelty weaves, will be processed.

John R. Roscow has been named manager of the printing department, under the direction of Charles R. Tagliabue, Vice-President in charge of the entire California plant. Mr. Roscow, who has been associated with United's Lodi, New Jersey printing department for a number of years, is thoroughly experienced in all phases of printing.

• Report on Synthetic Detergents

A comparative evaluation of certain synthetic detergents and descriptions of research work and plant processes for the production of some synthetic detergents are contained in one of three reports on German synthetic detergents and related products now on sale by the Office of Technical Services, Department of Commerce.

The 120-page report, prepared by four OTS investigators, John D. Branker, William H. Lockwood, Richard H. Nagel, and Kenneth L. Russell, also contains an alphabetical trade-name list of all synthetic detergents and most textile auxilieries made in Germany. Composition of the detergents and references to the source of information is also given.

The following classes of detergents are discussed separately in the report and the opinions of the German chemists, based mainly on laboratory tests but also on practical experience, are presented: alkyl sulfates, Igepons, Medialans, polyethylene oxide types, alkyl sulfonates, alkyl aryl sulfonates, and Lamepons.

Mimeograph copies of the report, PB-81819, *(Synthetic detergents and related surface active agents in Germany)* sell for $3.00. A supplementary report, PB-81819-S *(The P3 cleaners-Henkel & Cie., Dusseldorf;* 6 pages) sells for 25 cents mimeographed copy.

The third report, PB-80749, *(Action of alkali upon cellulose;* photostat, $6; microfilm, $2; 88 pages) is a translation of a research report prepared at the I. G. Farben Textile Application Library, Hoechst. The translation was made at the request of the Research and Development Branch of the Office of the Quartermaster General.

Orders for the reports should be addressed to the Office of Technical Services, Department of Commerce, Washington 25, D. C., and should be accompanied by check or money order, payable to the Treasurer of the United States.

• New German Reports

A comprehensive survey of German activity and developments in the fields of insecticides, insect repellents, rodenticides and fungicides is contained in a group of seven reports now on sale by the Office of Technical Services, Department of Commerce.

The seven reports, available in mimeograph form, comprise an almost complete account of German findings in these fields. The reports reveal that German developments were directed to the production of organic compounds which could be derived from materials other than such war short metals as lead, mercury, arsenic, copper, etc. As a result of research and development various organic substitutes were produced for the more common and formerly used inorganic chemicals.

The reports are:

PB-252 *(Insecticides, insect repellents, rodenticides and fungicides of I.G. Farbenindustrie, A. G., Elberfeld and Leverkusen, Germany;* mimeographed, 25 cents; 48 pages), reported by Dr. Stanley A. Hall of the Medical Intelligence Branch of the Office of the Chief Surgeon, Headquarters, U. S. Forces European Theatre.

PB-240 *(Manufacture of insecticides, insect repellents, rodenticides, I. G. Farbenindustrie A. G., Leverkusen and Elberfeld;* by Joseph E. Smadel and Francis J. Curtis; mimeographed, 25 cents; 12 pages) describes the preparation and method of use of some insecticides and related products produced at the Leverkusen and Elberfeld plants of I. G. Farben.

PB-306 *(Report on insecticide manufacturers in Hamburg;* by Dr. Stanley A. Hall; 2 pages; mimeographed, 10 cents) lists six insecticide manufacturers in the Hamburg area and gives information about the products they produce and the condition of their factories. All six manufacturers handled products containing well-known insecticides and none of them carried out research on new products nor did they have any experience with DDT or with insect repellents.

Related reports previously issued by OTS are:

PB-360 *(Insecticides and fungicides at the I. G. Farbenindustrie plant, Hoechst;* by Stanley A. Hall; mimeographed, 25 cents; 15 pages) discusses insecticides and fungicides produced at the Hoechst plant with particular attention given to GIX which contains as an active ingredient 60 percent 1, trichloro-2,2-bis (p-fluorophenyl) ethane.

PB-22413 *(Investigation of insecticide and insectifuge research and manufacture in Western Germany;* by Lowell B. Kilgore; mimeographed, 75 cents; 25 pages) reports the results of an investigation to obtain all information available on the development, manufacture and use of insecticides, insectifuges, fungicides, and rodenticides in Western Germany.

PB-60890 *(Organic chemical intermediates for insecticides, fungicides and rodenticides;* mimeographed, $1.25; 44 pages) gives manufacturing details for the production of organic chemicals used in compounding insecticides, fungicides, and rodenticides in Germany. The report was prepared by J. T. Thurston, director, chemical research laboratories, American Cyanamid Co., Stamford, Conn., who investigated the German industry under OTS sponsorship.

PB-77670 *(Fluorobenzene manufacture;* by F. O. Robitschek and B. H. Wilcoxon; mimeographed, 50 cents; 13 pages) covers the manufacturing procedure for the preparation of fluorobenzene at the Hoechst plant, the only German plant producing this ingredient for GIX.

Orders for the reports should be ad-

• Joins Mathieson Chemical

Dr. D. P. Morgan who has joined the Mathieson Chemical Corporation, formerly The Mathieson Alkali Works, as Administrator of Research and Development. Dr. Morgan will be in charge of market research as well as chemical research and development. For the past two years he has been with W. R. Grace and Company in New York in charge of chemical market research and development.

Dr. Morgan was graduated from Harvard University in 1916 and received a Ph.D. in chemistry from Columbia University in 1923. He was a partner in the firm of Loomis, Stump and Banks, consulting chemical engineers, for four years before joining Scudder, Stevens and Clark as chemical consultant. For the last several years of the war he served as Director of the Chemicals Bureau of the War Production Board.

A fellow of the American Institute of Chemists, Dr. Morgan is also a member of many other organizations, including the American Chemical Society, the American Institute of Chemical Engineers, and Sigma Xi honorary scientific society.

dressed to the Office of Technical Services, Department of Commerce, Washington 25, D. C., and should be accompanied by check or money order payable to the Treasurer of the United States.

• General Aniline Safety Record

The dyestuffs plant of General Aniline & Film Corporation at Grasselli, N. J., has established a plant safety record of 1,500,000 man hours of work without a single lost-time accident, Plant Manager Russell I. Baker announced recently. The accident-free period in this plant of 2,300 employees covers the four months from December 12th, 1947 to April 10th.

The record is the result of the joint efforts of the Company and its employees, and is a measure of the steady gain in accident prevention since the adoption five years ago of the present integrated safety program.

The new record may be reviewed in the light of the 1947 New Jersey Inter-plant Safety Contest in which the competing industries averaged 18 lost-time accidents per plant over a similar period.

All General Aniline employees are provided with safe practice handbooks pertinent to plant operations. Rotating safety committees of employees are appointed monthly in all departments and safety meetings are held every month in each department for open discussion of accident prevention problems.

• Lubricity Tester

The prime purpose of the new Lubricity Tester, developed by the Nopco Chemical Company, Inc., and built by the United States Testing Company, is the determination of friction qualities of yarn. The equipment utilized, measures the speed of a rider down a known incline for a fixed distance The test apparatus is applicable to filament yarns and threads.

It is stated that the method of test very closely duplicates actual running conditions so that actual effects in relative use are reflected in results. The Lubricity Tester has been used in the evaluation of knitting lubricants on various yarns and in comparison of sewing threads.

The Lubricity Tester may be used in determining the effect of mercerization on the smoothness of the yarn, relationship between coefficient of friction and abrasion resistance in processing, and in other fields.

• Moisture Register

Announcement of a new model pressurized Moisture Register, designed to accurately test moisture content of loose and compressible materials used in the textile industry, has just been made by Moisture Register Co., 133 N. Garfield Ave., Alhambra, California. This instrument is now being specifically used on scoured wool, combed wool (tops), wool yarns (skeins), wool blankets, and bulk cotton.

Identified as Moisture Register Model PD-1, it is the newest in a series of electronic moisture meters now available, after thorough field testing in actual use.

It is stated that, through deep penetration by high frequency field, it tends to average moisture content of material to a depth of three inches as compared to a penetration heretofore limited to a depth of one inch.

Complete details are available without obligation, upon request to the manufacturer. Since there is a design variation between instruments for cotton and woolen materials, type of material to be tested should be specified.

• Research Director

Leonard Shapiro, who has been appointed Research Director of Synthron, Inc., textile specialty manufacturers of Ashton, R. I. Robert Zametkin, President of Synthron, announced that the rapid growth of this relatively young company has necessitated the construction of a new laboratory to be devoted entirely to research and development aimed at the completion of Synthron's line of textile auxiliaries.

Leonard Shapiro is well-known in the textile industry for his work on textile applications as well as chemical research and development. In his former capacity as Textile Research Director for Alrose Chemical Co., he was intimately associated with the development of the well-known Definized Process for dimensional stabilization of rayon and the Protonized Process for shrinkage-control of wool. He was formerly Director of Textile Research at Warwick Chemical Co., where he was best known for his work on textile resins and durable water repellents. He also spent many years in the coated fabrics industry as Director of Laboratories at Interlaken Mills, and was Research Assistant to Prof. E. R. Schwarz at the M.I.T. Textile Laboratory. He received his B.S. and M.S. degrees at M.I.T. He is the author of numerous publications in the fields of textile microscopy, textile calculations, resin finishes, and dimensional stabilization.

• Reports on Microbiological Degradation

A high degree of resistance to microbiological degradation can be imparted to cotton cloth through impregnation with various resins according to a report now on sale by the Office of Technical Services, Department of Commerce.

The report is one of a series prepared by the Tropical Deterioration Research Laboratory of the Office of the Quartermaster General as a result of its research on the development of a microbiologically resistant fabric. Three other reports of the series are also available from OTS.

The latest report in the series, PB-86252, *(Resin impregnation of cotton fabrics for prevention of microbiological decay;* by Drs. W. Lawrence White and Ralph G. H. Siu; mimeographed, 50 cents; 9 pages) is concerned with the use of resin impregnations as an inert physical barrier between the invading microorganisms and the cotton fiber.

The other three reports in the series are:

PB-31871 *(Field studies of the deterioration of textiles under tropical conditions;* by Elso S. Barghoorn; microfilm, $6; photostat, $18; 268 pages).

PB-22339 *(The microbiological degradation of cotton fabrics;* by Ralph G. H. Siu and W. Lawrence White; photostat, $5; microfilm, $1; 63 pages).

PB-22340 *(The effect of ultraviolet light on cotton cellulose and its influence on subsequent degradation by micro-organisms;* by R. P. Wagner, Harold H. Webber, and Ralph G. H. Siu; photostat, $6; microfilm, $1; 65 pages).

Orders for the reports should be addressed to the Office of Technical Services, Department of Commerce, Washington 25, D. C., and should be accompanied by check or money order payable to the Treasurer of the United States.

• Director, AICE

John J. Healy, Jr., Assistant General Manager, Merrimac Division, Monsanto Chemical Company, has been elected a Director of the American Institute of Chemical Engineers for the next three years.

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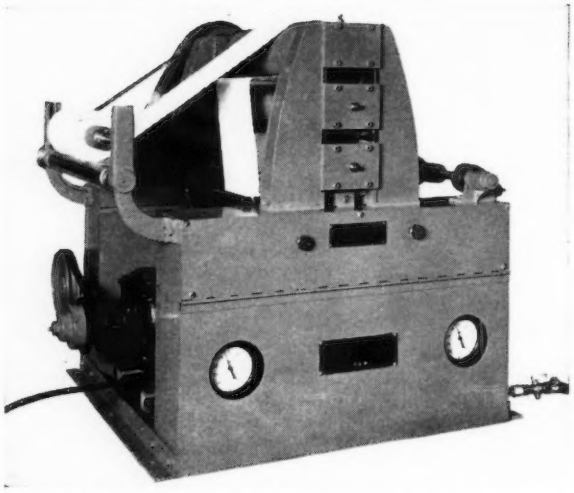

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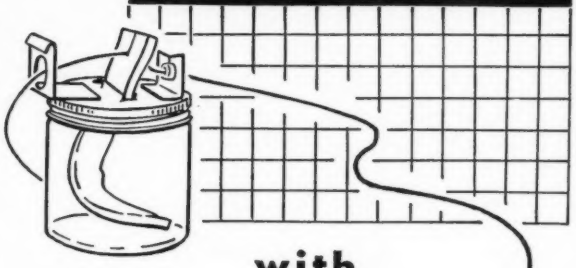

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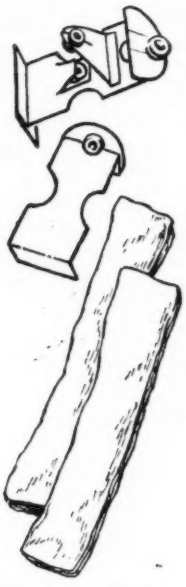

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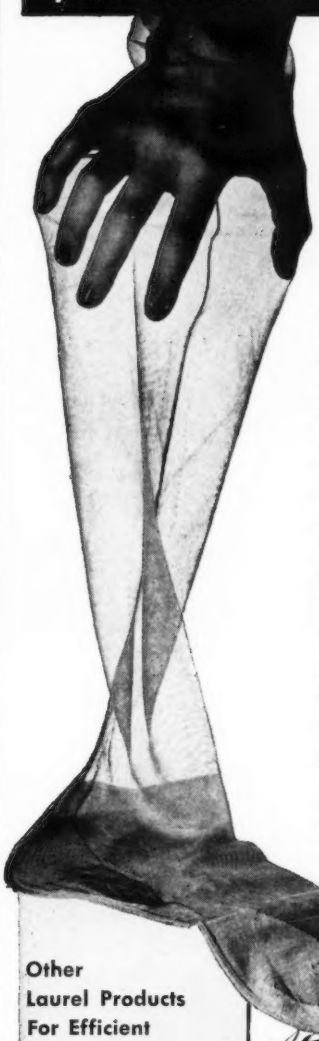

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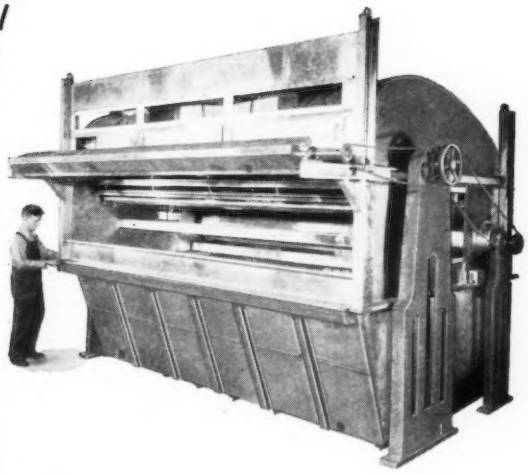

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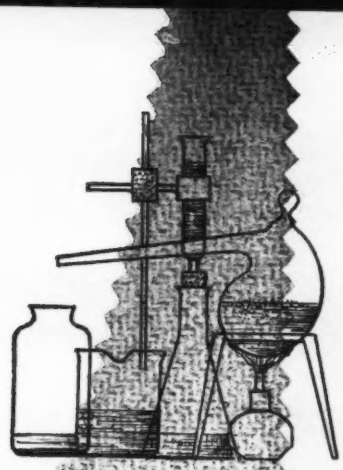

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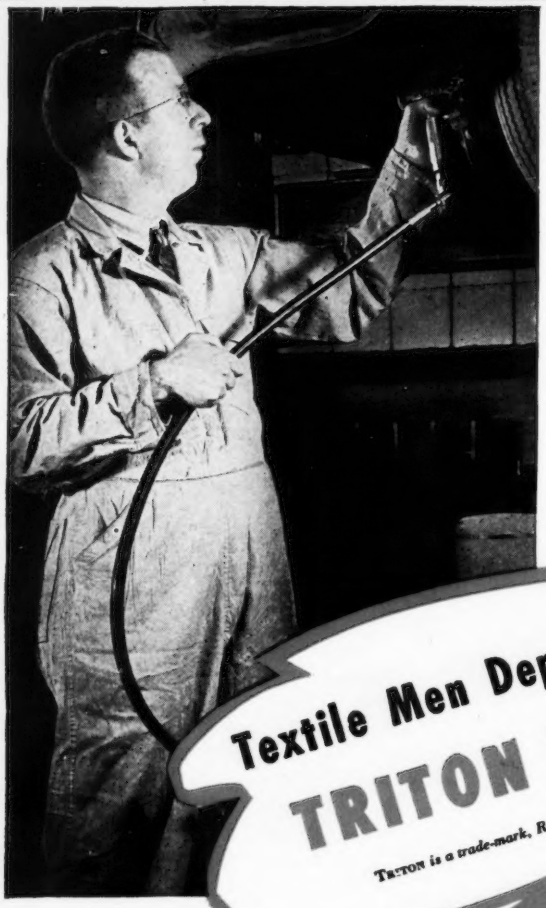

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